AEROSPACE REPORT NO.
TOR-2015-00876

DebriSat Laboratory Analyses

January 5, 2015

Paul M. Adams[1], Zachary Lingley[2], Nathan Presser[2], and Gouri Radhakrishnan[1]
[1]Space Materials Laboratory, Physical Sciences Laboratories
[2]Electronics and Photonics Laboratory, Physical Sciences Laboratories

Prepared for:

Space and Missile Systems Center
Air Force Space Command
483 N. Aviation Blvd.
El Segundo, CA 90245-2808

Contract No. FA8802-14-C-0001

Authorized by: Space Systems Group

Report Documentation Page

Form Approved
OMB No. 0704-0188

1. REPORT DATE	2. REPORT TYPE	3. DATES COVERED
05 JAN 2015	**Final**	**-**

4. TITLE AND SUBTITLE	5a. CONTRACT NUMBER
DebriSat Laboratory Analyses	**FA8802-14-C-0001**
	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)	5d. PROJECT NUMBER
Paul M. Adams, Zachary Lingley, Nathan Presser, and Gouri Radhakrishnan	5e. TASK NUMBER
	5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)	8. PERFORMING ORGANIZATION REPORT NUMBER
The Aerospace Corporation 2310 E. El Segundo, CA 90245-4609	**TOR-2015-00876**
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S)
Space and Missile Systems Center Air Force Space Command 483 N. Aviation Blvd. El Segundo, CA 90245-2808	**SMC**
	11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release, distribution unlimited

13. SUPPLEMENTARY NOTES
The original document contains color images.

14. ABSTRACT
The DebriSat test was conducted to better understand the distribution of fragments generated from a hypervelocity impact with a modern satellite. The last such test (SOCIT) was conducted 20 years ago and satellite construction has changed considerably since then. DebriSat was a NASA program with support/collaboration from the Air Force Space and Missile Center, University of Florida and Aerospace. Tests were conducted at the Arnold Engineering Development Complex Range G Two-Stage Light Gas Gun Facility. The 50 kg target was a constructed by the University of Florida from materials representative of a modern LEO satellite. The Aerospace Concept Design Center advised on the selection of materials for various subsystems.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			
unclassified	**unclassified**	**unclassified**	**UU**	**106**	

Abstract

The DebriSat test was conducted to better understand the distribution of fragments generated from a hypervelocity impact with a modern satellite. The last such test (SOCIT) was conducted 20 years ago and satellite construction has changed considerably since then. DebriSat was a NASA program with support/collaboration from the Air Force Space and Missile Center, University of Florida and Aerospace. Tests were conducted at the Arnold Engineering Development Complex Range G Two-Stage Light Gas Gun Facility. The 50 kg target was a constructed by the University of Florida from materials representative of a modern LEO satellite. The Aerospace Concept Design Center advised on the selection of materials for various subsystems. The test chamber was lined with "soft catch" foam panels to trap fragments for size distribution analysis. A witness plate assembly was constructed by Aerospace in order to catch and sample debris and returned to Aerospace after the test for analysis. Aerospace also placed SEM stub witness plates into the soft catch panels for post test retrieval and analysis. The test was conducted with a pressure of 1-2 Torr of air and used a ~600 gram projectile with a nominal velocity of 7 km/s.

The SEM stubs, witness plate assembly and DebriSat fragments were covered with black soot and were contaminated with soft catch foam fragments. The SEM stubs were also covered with a thin film of condensed soft catch vapor, similar to that seen with the Debris-LV test which also used soft catch foam panels. Deposits on the SEM stubs and witness plate assembly are predominantly carbon and consist of agglomerates of nano carbonaceous material. These deposits are primarily from the soft catch (similar to Debris-LV) though the C-C composite honeycomb face sheets and MLI are also possible sources. Disordered graphitic carbon is present based on Raman spectra and TEM lattice fringe images. Graphitic carbon was also seen in Debris-LV deposits even though there were no carbon containing materials in the target. The witness plate assembly was covered in a black layer of loose "soot", even under the protective Whipple plates. Removal of the loose material by rinsing with isopropyl alcohol revealed a thin adherent coating on one side of the support posts indicating early directional deposition from DebriSat. The coating was carbonaceous (disordered graphite) with nano metal droplets. Fluorine from Teflon wire insulation was also common in the SEM stub and witness plates deposits. Nano droplets of metallic materials (Al, Fe, Cu, Zn, Ge) were also present indicating melting as a result of the impact. Solidified molten metal droplets were also seen in Pre Preshot and Debris-LV debris. Aluminum was from the Al honeycomb, nadir and zenith panels, structural core and COPV liner. Aluminum oxide particles were also present. Iron was from stainless steel tubing and solenoids. Germanium was from the solar cells and copper was from wiring and solenoids. The source of the zinc has not been identified. The solidified molten nano metal droplets are crystalline based on TEM lattice fringes and consist of only a few crystallites.

The witness plates show a significant decrease in reflectance (95% to 6%). Soft catch contamination was seen in the LWIR reflectance spectra of DebriSat fragments, SEM stubs and witness plate. As a result, it was not possible to get a clean spectrum of the debris generated by the hypervelocity impact. An additional "oxide" band, which may be from a form of aluminum oxide, was seen on some samples.

Acknowledgements

DebriSat Team Members:

J.-C. Liou: NASA Space Debris Program Office, NASA JSC
AEDC Range G Light Gas Gun Staff
Charles Griffice: Aerospace
Marlon Sorge: Aerospace
Patti Sheaffer: Aerospace

Financial Support:

Charles Griffice and Tom Huynh

Discussions with:

Patti Sheaffer
Charles Griffice

UV-VIS-NIR Spectroscopy

Dianna Alaan

AEROSPACE
Assuring Space Mission Success

Aerospace Technical Operating Report No.
TOR-2015-00876

DebriSat Laboratory Analyses

5 January, 2015

Paul M. Adams[1], Zachary Lingley[2], Nathan Presser[2] and Gouri Radhakrishnan[1]

[1]Materials Processing Department
Space Materials Laboratory
[2]Microelectronics Technology Department
Electronics and Photonics Laboratory

Physical Sciences Laboratories

Introduction

- The DebriSat test was conducted to better understand the distribution of fragments generated from a hypervelocity impact with a modern satellite.
  - The last such test (SOCIT) was conducted 20 years ago and satellite construction has changed considerably since then.
  - In 2009 a Cosmos 2251 upper stage collided with an Iridium 33 satellite.
    - Produced 2000+ trackable fragments (>10 cm).
  - 8 other known collisions, some only known long after occurrence.
- DebriSat was a NASA program with support/collaboration from the Air Force Space and Missile Center, University of Florida and Aerospace.
- Tests were conducted at the Arnold Engineering Development Complex, Tullahoma, Tennessee.
  - Two-Stage Light Gas Gun Facility - Range G.
  - Largest such facility in the United States.
  - All tests used a ~600 gram projectile with a nominal velocity of 7 km/s.

Introduction (cont.)

- Two trial tests were conducted prior to DebriSat.
  - Pre Preshot. February 2014
  - Debris-LV (Pre Shot). 1 April 2014
- Debris-Sat was conducted 15 April 2014
  - The 50 kg target was constructed by the University of Florida from materials representative of a modern LEO satellite.
    - Aerospace Concept Design Center advised on selection of materials for various subsystems.
  - Test chamber was lined with “soft catch” foam panels to trap fragments for size distribution analysis.
  - A witness plate assembly was constructed by Aerospace in order to catch and sample debris and returned to Aerospace after the test for analysis.
  - Aerospace also placed SEM stub witness plates into soft catch for post test retrieval and analysis.
  - Test conducted with a pressure of 1-2 Torr of air.

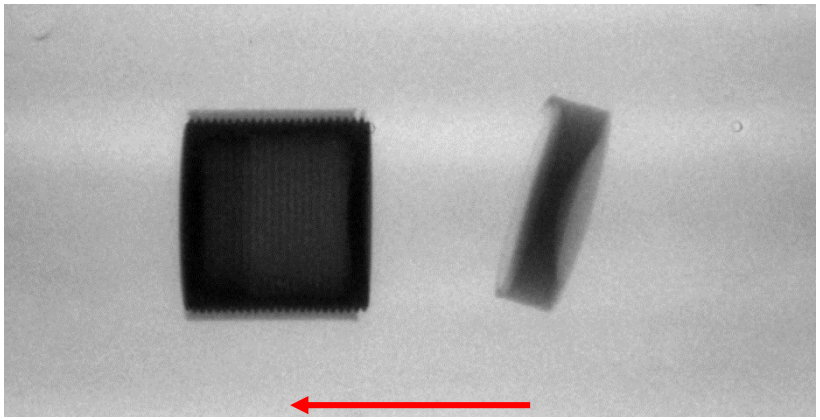
Introduction (cont.)

- Documentation to date.
  - Aerospace TOR-2014-03201, Time-resolved Spectroscopy of Hypervelocity Impact Flash on DebriSat, Gouri Radhakrishnan.
  - Aerospace ATM-2014-03659, DebriSat Hypervelocity Impact Fragmentation Modeling, Naoki Hemmi.

- Background
  - “Darkening” of satellites has been observed as a result of suspected hypervelocity impacts.
  - The material and processes responsible for the darkening is unknown.
- Objectives
  - Materials collected on witness plates in the DebriSat test were analyzed in order to identify the source and conditions responsible for the darkening.
  - UV-VIS-NIR-LWIR reflectance spectra were measured of post test debris for comparison with pre test sources to determine the spectral signature of material generated by a hypervelocity impact.
  - Possibly determine if a hypervelocity impact occurred based on remotely sensed spectra?
    - Can the source be identified – does it have a unique signature?

Projectile

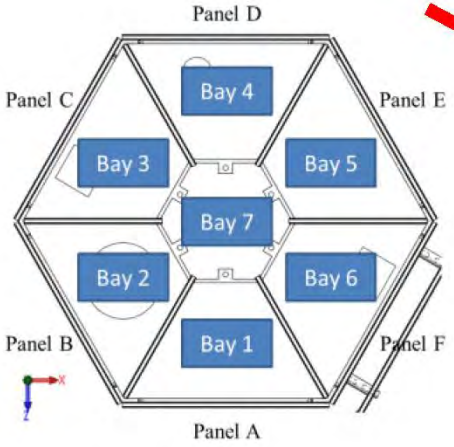
Images by AEDC



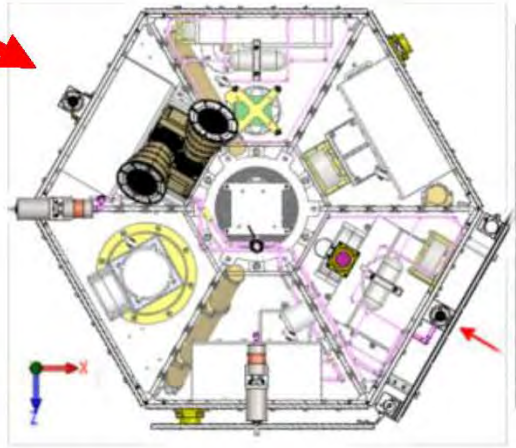
Flash X-ray of projectile in flight

- Constructed from three pieces: Outer Nylon shell (sabot) with 2 part hollow aluminum insert.
- ~600 grams, 8.6 cm diameter X 10.3 cm long – size of a soup can.
- Velocity ~ 6.8-6.9 km/s.
- The Nylon base separated from the Nylon-aluminum body during flight.

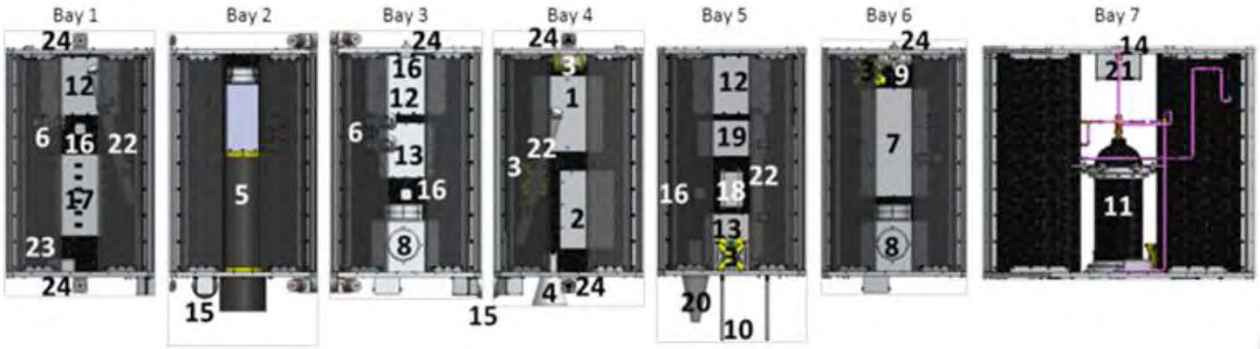
DebriSat Construction



Impact



Component Identification	
1 – Flight Computer	13 – Telemetry Avionics (2)
2 – Data Recorder	14 – Fill Drain Valve
3 – Reaction Wheel (4)	15 – Star Tracker (2)
4 – X-Band Conical Feed Horn Antenna	16 – Sun Sensor (4)
5 – Optical Imager	17 – Payload Support Module
6 – Solenoids (3)	18 – Magnetometer
7 – Power Conditioning and Distribution	19 – ADCS Avionics
8 – Spectrometer (2)	20 – S-Band Helical Antenna
9 – Thermal Reservoir	21 – Propulsion Avionics
10 – Omni-Directional Antenna (2)	22 – Magnetorquer (3)
11 – COPV	23 – Inertial Measurement Unit
12 – Li-ion battery box (3)	24 – Thrusters (6)



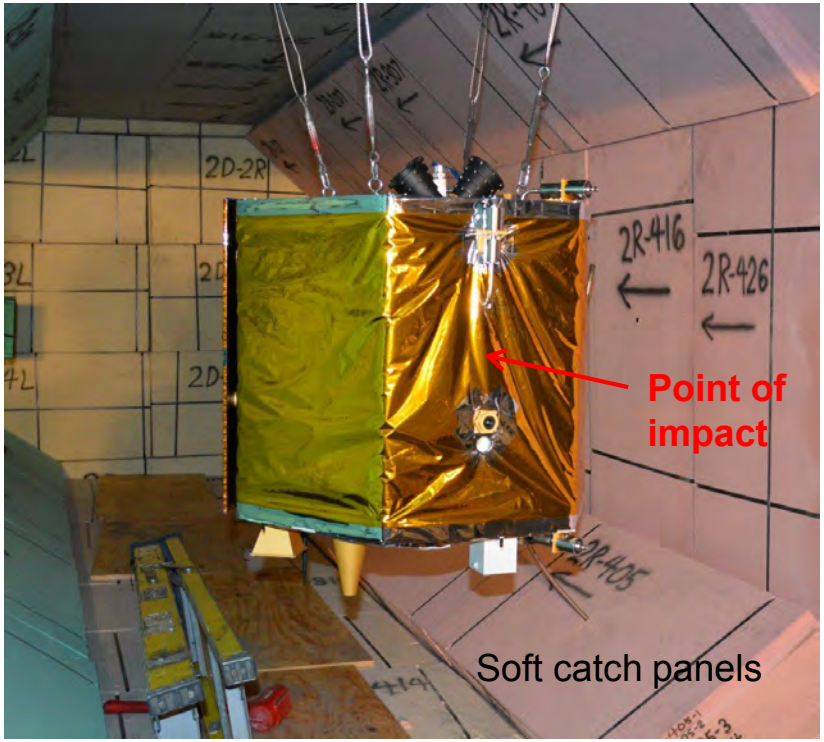
Projectile impacted normal to Bay 3

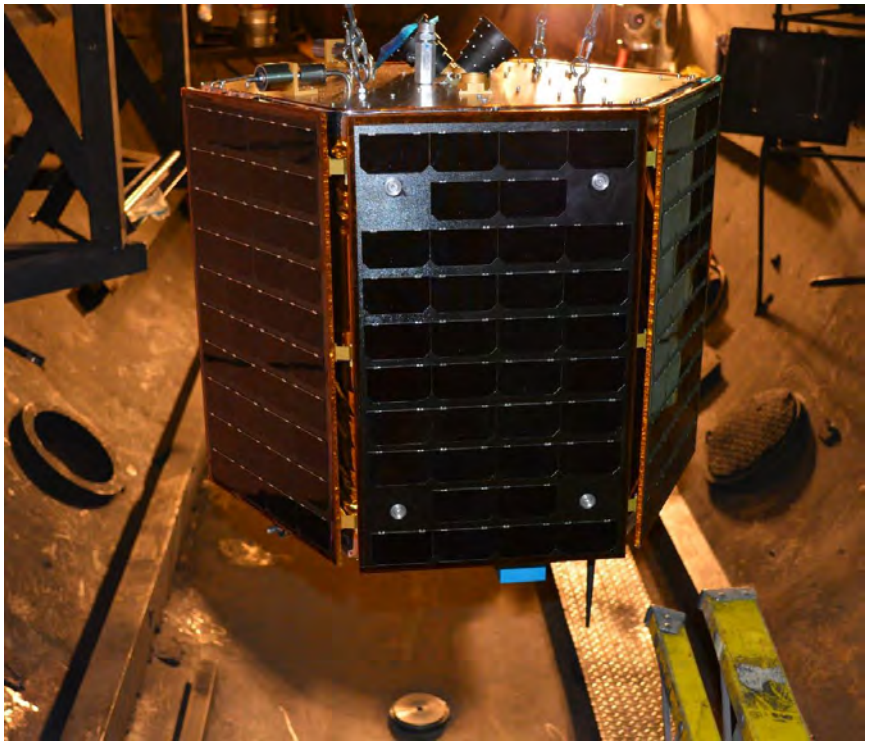
Figures from U. of Florida

Materials Present on DebriSat

- Nadir and zenith panels: Aluminum 6061
- Face panels and structural ribs: Aluminum 5052 honeycomb with carbon fiber/epoxy face sheets
- COPV tank pressurized to 2 torr with air
- Optics : SiO_2, sapphire
- Solenoids : Cu
- Stainless Steel (316, 304) : Fe, Cr, Ni
- Printed circuit boards (electronics hardware)
- Li-ion batteries: Cu-polyimide (no Li or electrolyte)
- Solar cells: Ge substrate with GaAs/InP/GaInP
- Kevlar, polyurethane, Mylar, Kapton

Installed in Chamber: Pre test

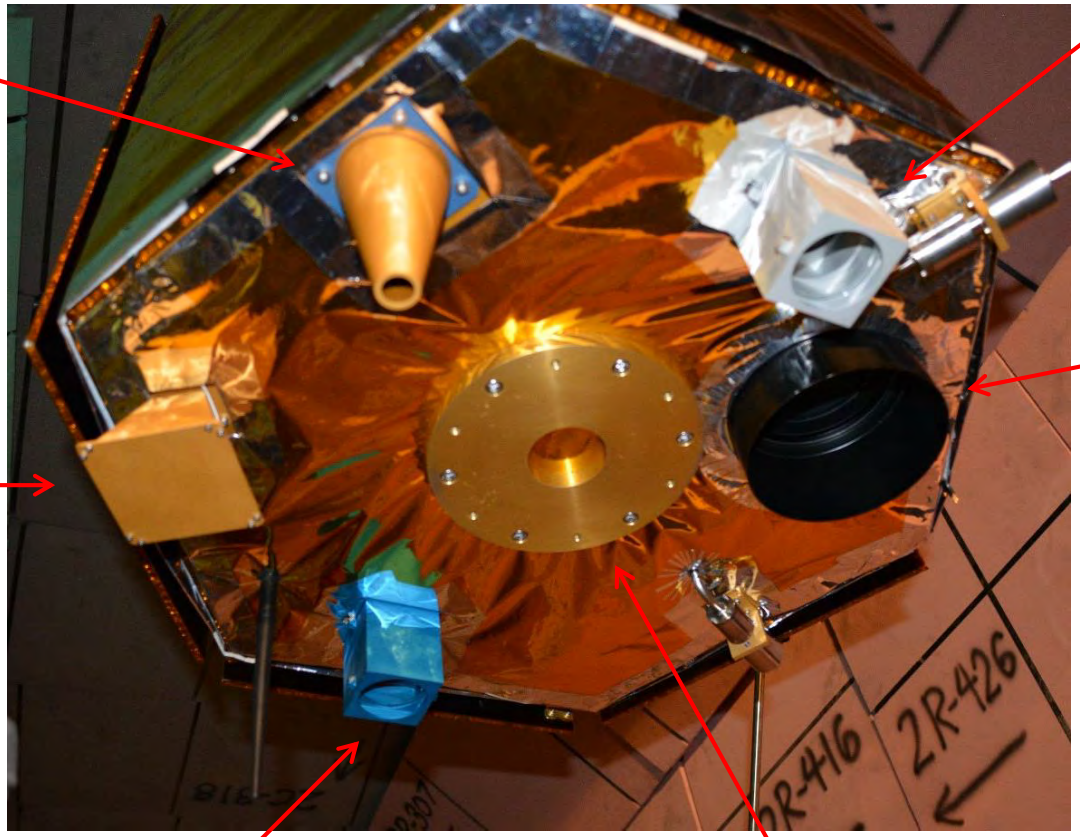




Looking down range.
DebriSat is covered with multi layer insulation (MLI).

Looking up range.
Solar panels - undeployed

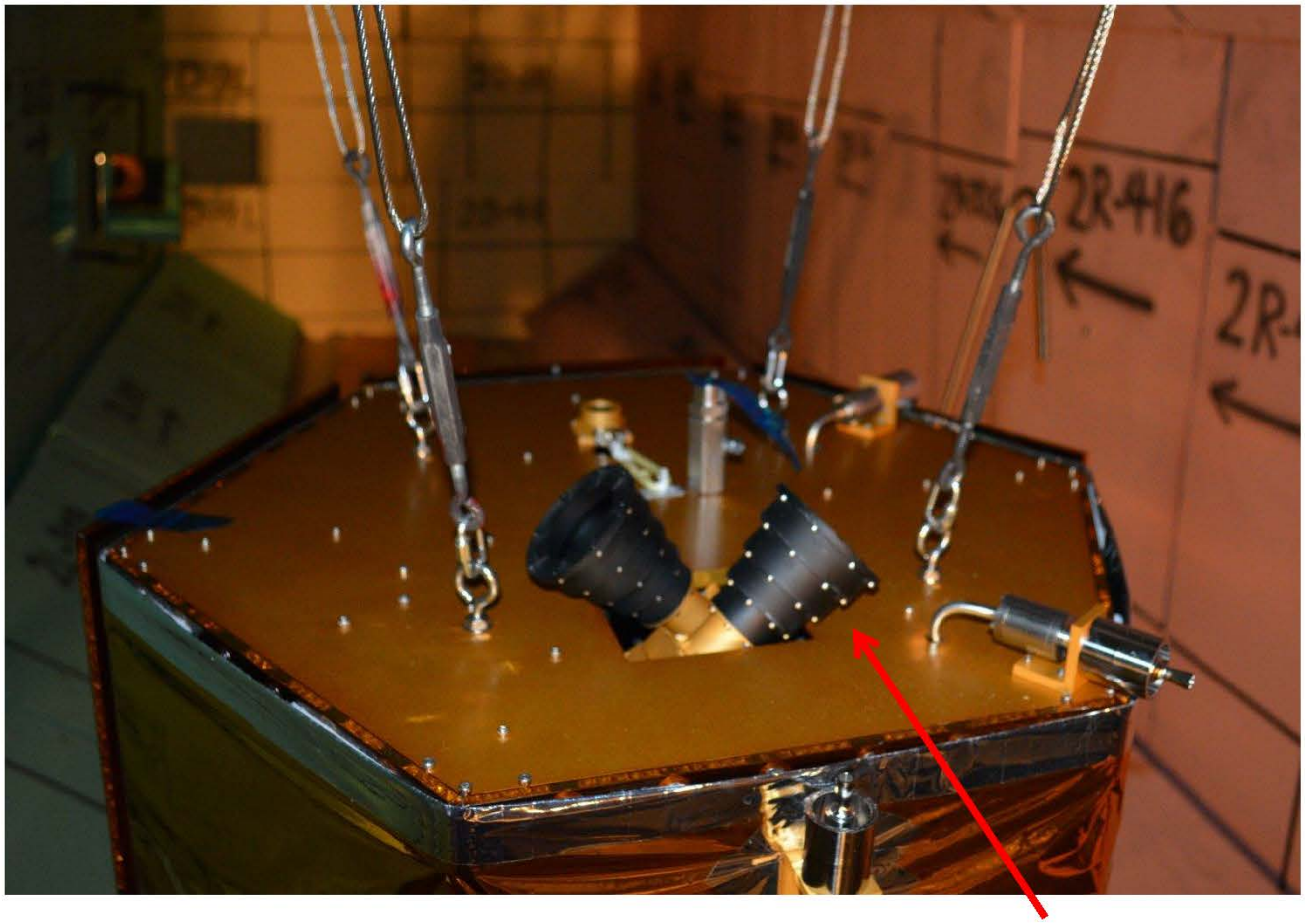
Nadir / Under Side



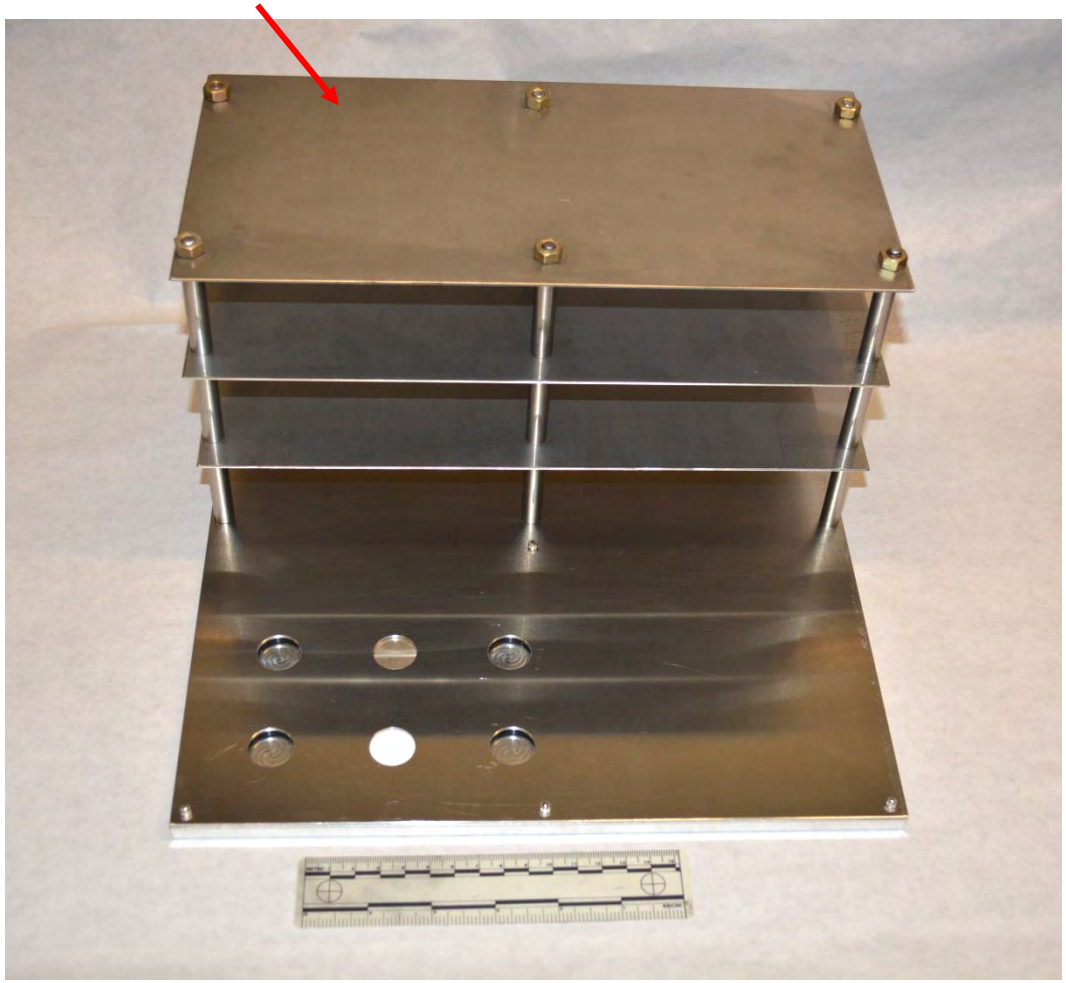
Aluminum components had various types of anodized finishes (clear, black, blue, gold) to aid in post test fragment identification.

SBU Marking

Zenith / Top Side



Witness Plate: Pre Test



Witness Plate Samples:

<u>Direct Exposure</u>

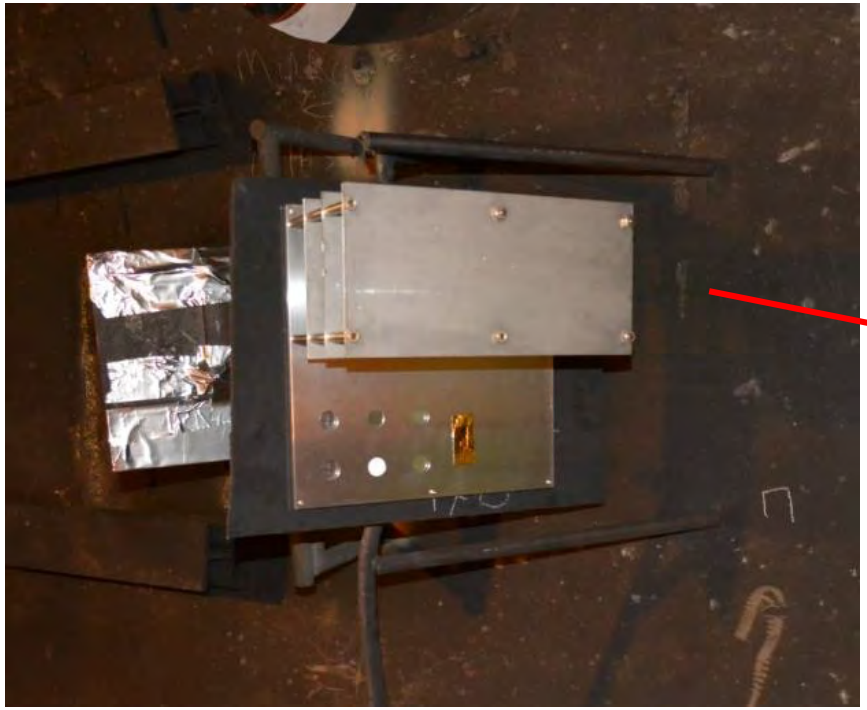
(4) 1” fused silica
(1) 1” Z-93 painted Al
(1) 1” Aluminum
Multi layer insulation (not shown)

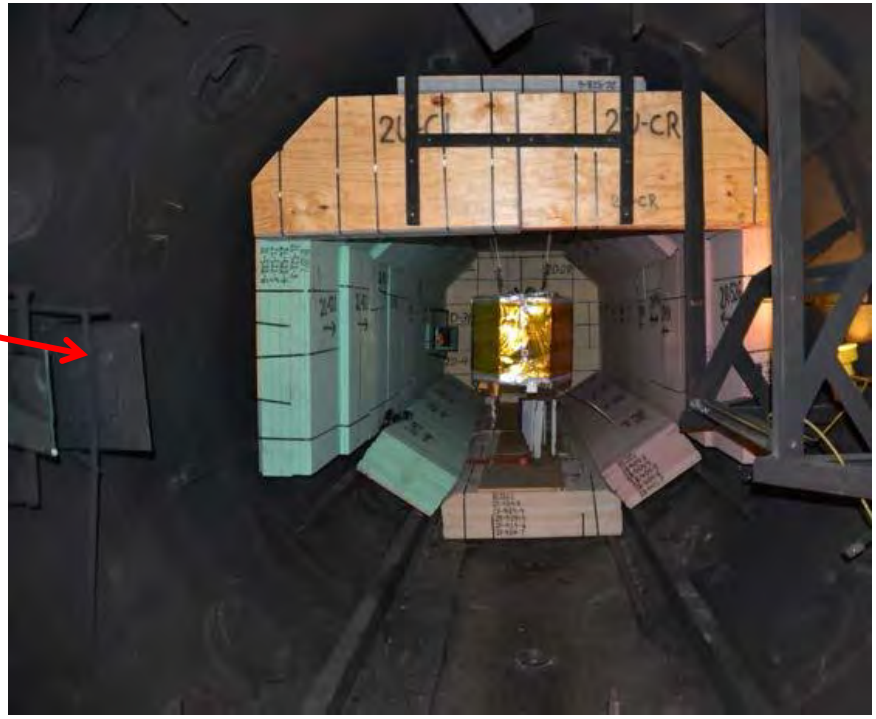
<u>Protected Under Whipple Plates</u>

(2) 1” fused silica
(1) 1” Z-93 painted Al
(1) 1” Aluminum
(1) 1” NaCl
(1) Cu sheet
Ge ATR crystal (for FTIR)
Solar cell

Witness plates located in same position in chamber as Debris-LV.
~3 meters up range of DebriSat.

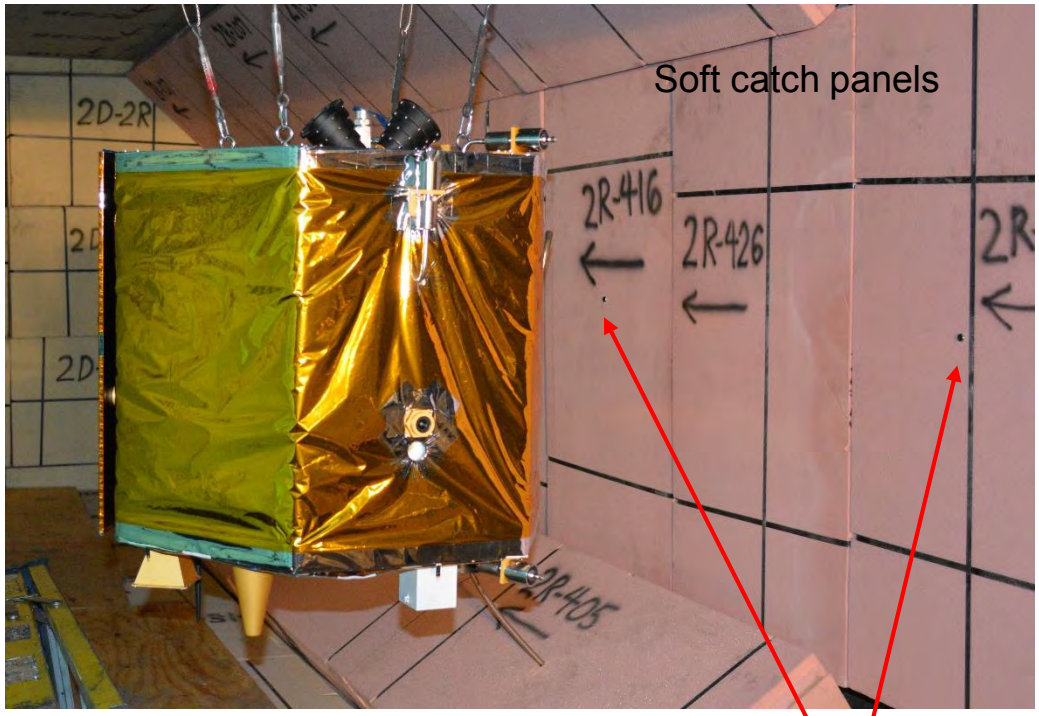
Witness Plates Mounted in Chamber



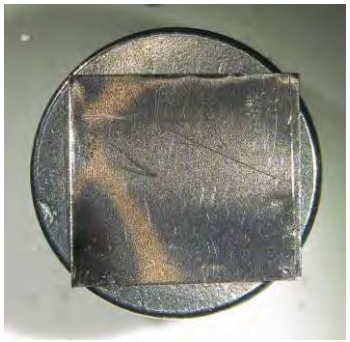


Plates mounted about 3 meters up range of DebriSat

SEM Stub Witness Plates Placed into Soft Catch Panels



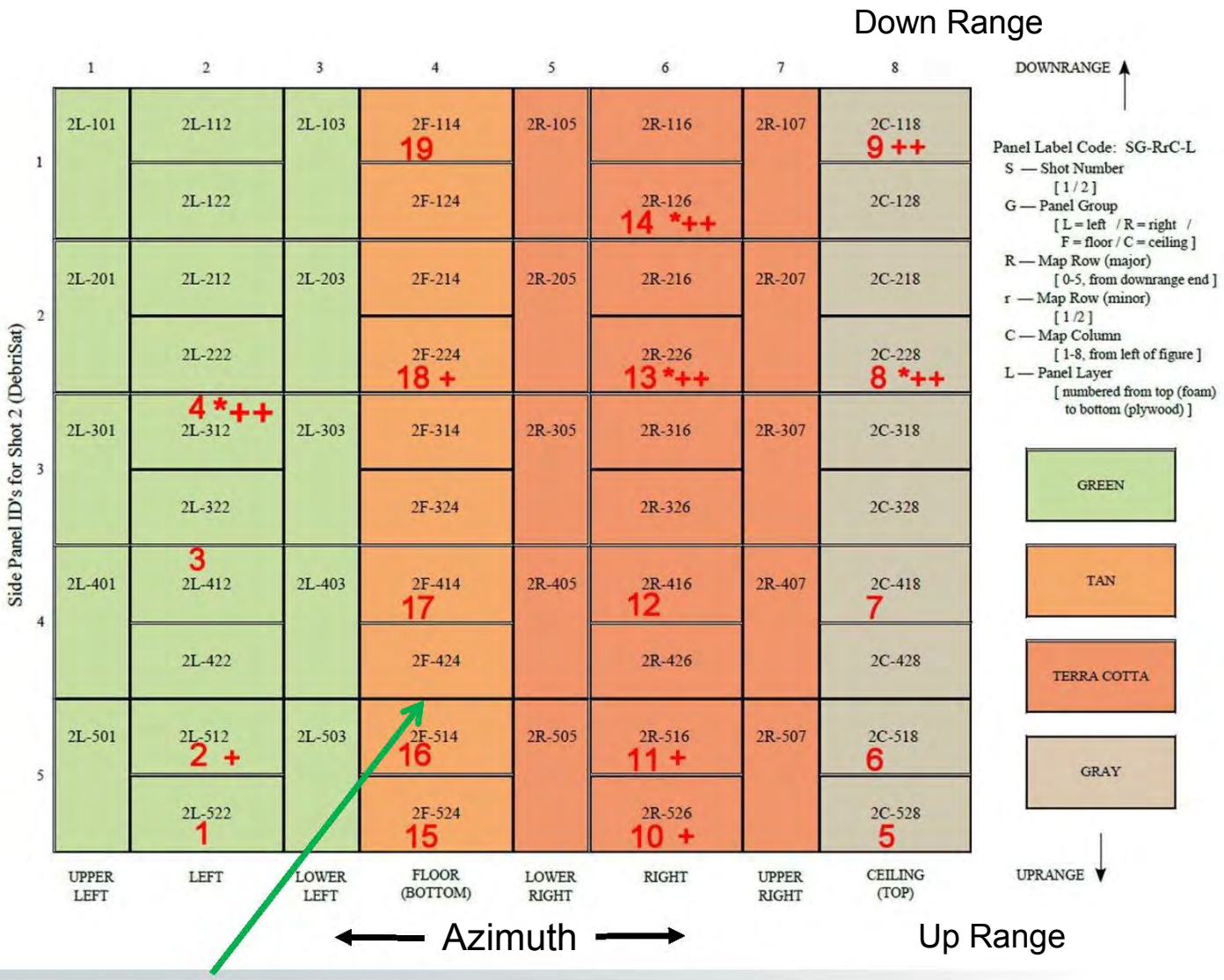
12.5 mm Al SEM stubs



with Ta sheet

- 24 Aluminum SEM stub witness plates (12.5 mm dia) placed in soft catch with RTV adhesive.
- Tantalum sheet (9 mm x 9 mm) epoxied to front surface in order to distinguish Al debris from stub.
- Identified on back with engraved numeral.
- 11 stubs recovered (+); 5 with Ta sheet present (++), 6 in place (*)
- Remaining stubs embedded in soft catch. – to be recovered at U. of F.

SBU Marking
Location of SEM Stubs (red) on Soft Catch Panels



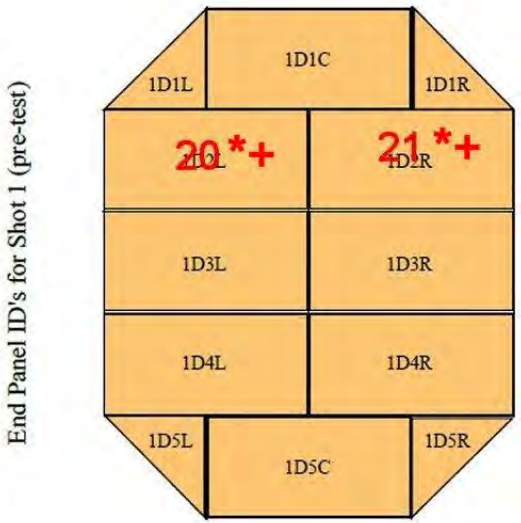
Nominal location of DebriSat is above 2F-424 and 2F-514

stubs recovered (+), with Ta sheet present (++), in place (*)

Intact recovered stubs with Ta (*++) tended to be furthest from impact site

Location of SEM Stubs (red) on Soft Catch Panels

Down Range End Panels



DOWNRANGE
(looking downrange)

Panel Label Code: SERC-L
S — ShotNumber [1/2]
E — PanelGroup [D=downrange]
R — MapRow [1-4, from top of figure]
C — MapColumn [L=left / R=right / C=center]
L — PanelLayer [numbered from top (foam) to bottom (plywood)]

Upper Up Range End Panels

Lower Up Range End Panels

UPRANGE
(looking downrange)

Panel Label Code: SEGC-L
S — ShotNumber [1/2]
E — PanelGroup1 [U=uprange]
G — PanelGroup2 [F=floor / C=ceiling]
C — MapColumn [L=left / R=right]
L — PanelLayer [numbered from top (foam) to bottom (plywood)]

•stubs recovered (+), with Ta sheet present (++), in place (*)

DebriSat Impact: Frames from High Speed Video

T = -180 μS

T ~ 0 μS

High speed camera (65,000 fps) images from NASA

DebriSat Impact: Frames from High Speed Video

T = +45 μS

T = +270 μS

High speed camera (65,000 fps) images from NASA

Laboratory Results

(Supplemental Information and Additional Analyses in Appendix)

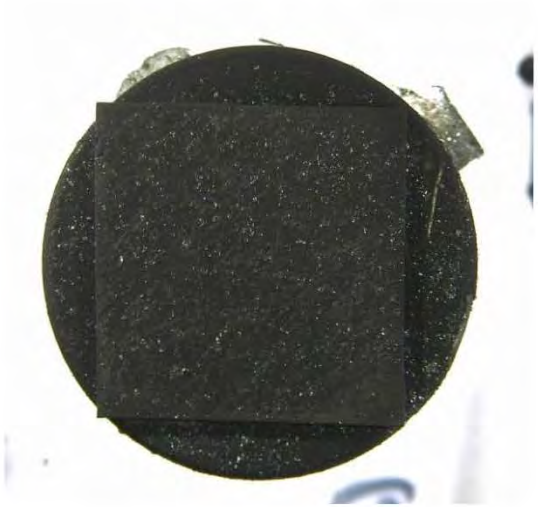
Laboratory Methods

- Scanning Electron Microscopy (SEM)
  - High resolution imaging.
  - Atomic number contrast.
- Transmission electron microscopy (TEM)
  - Ultra high resolution – lattice imaging (crystallinity)
  - Electron diffraction – crystallinity – phase identification
- Energy Dispersive (X-ray) Spectroscopy (EDS) in the SEM/TEM
  - Semiquantitative elemental composition.
  - Elemental mapping and line scans.
- Fourier Transform Infrared (FTIR) spectroscopy
  - Identification of chemical functional groups.
  - Correlation with LWIR hyperspectral remote sensing signatures.
- Raman Spectroscopy
  - Identification of forms of carbon
- UV-VIS-NIR Spectroscopy
  - Measurement of darkening at UV-VIS-NIR wavelengths.

SEM Stubs (post test)

8

9

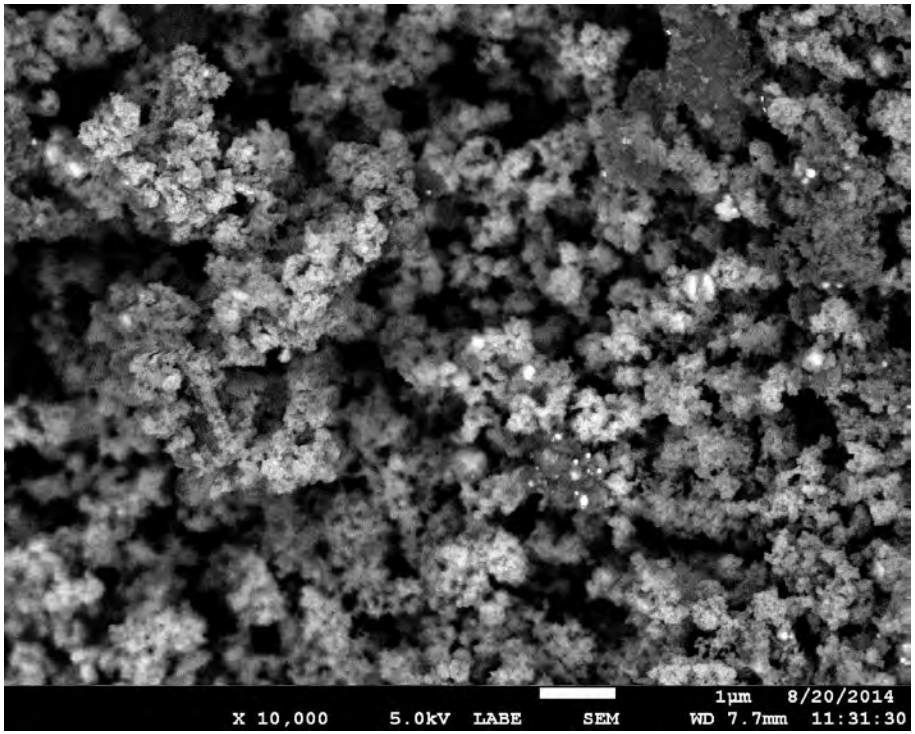


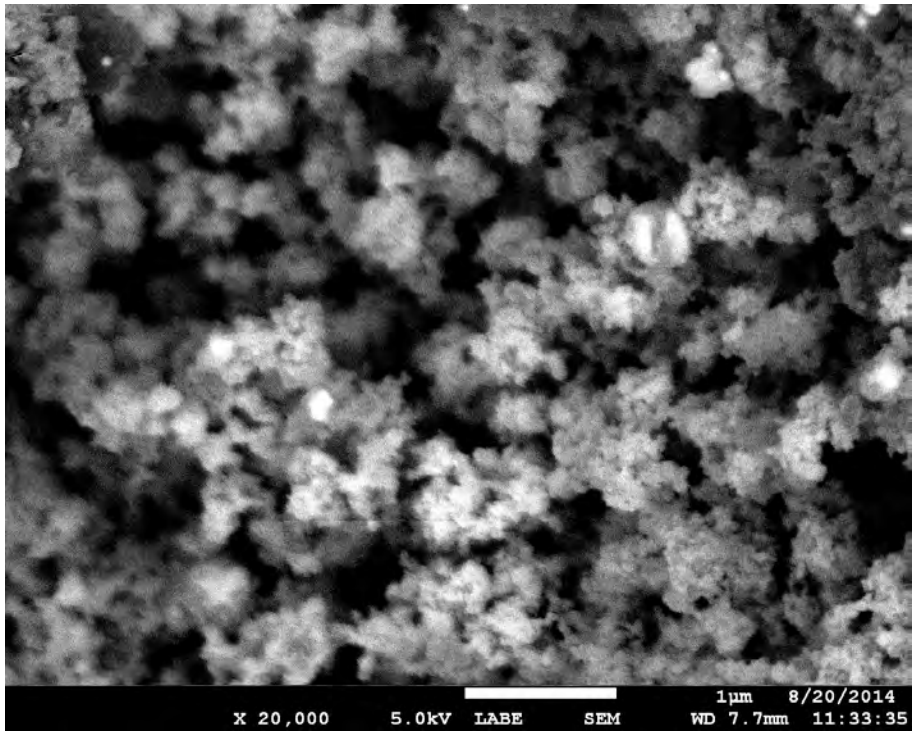
13

14

Stubs are covered with a black soot-like substance

SEM Stub 4:
Backscatter Electron SEM (10KX, 20KX)



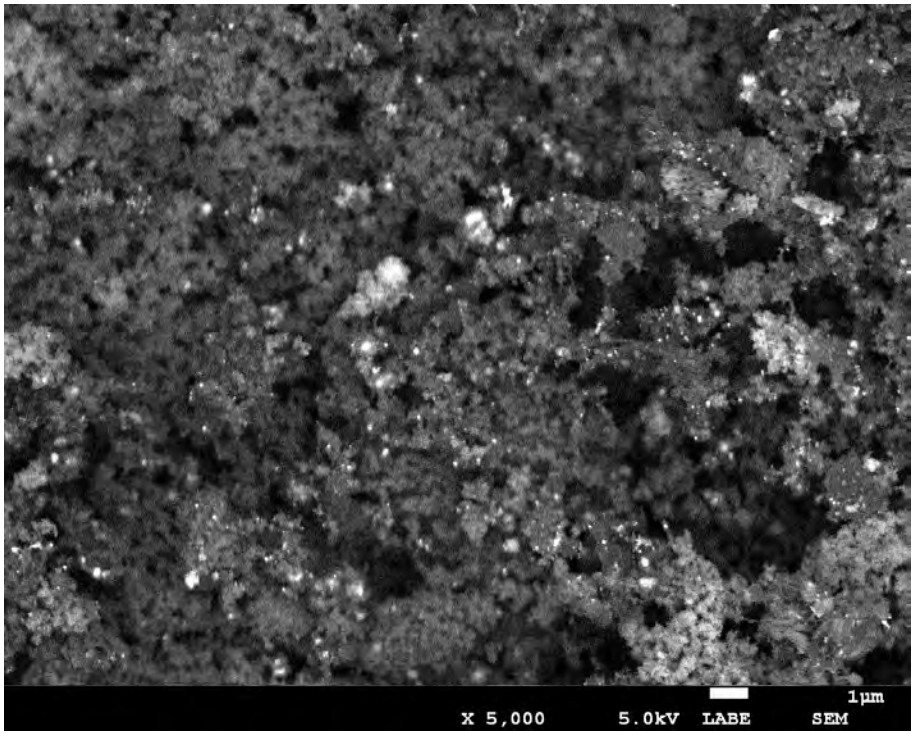


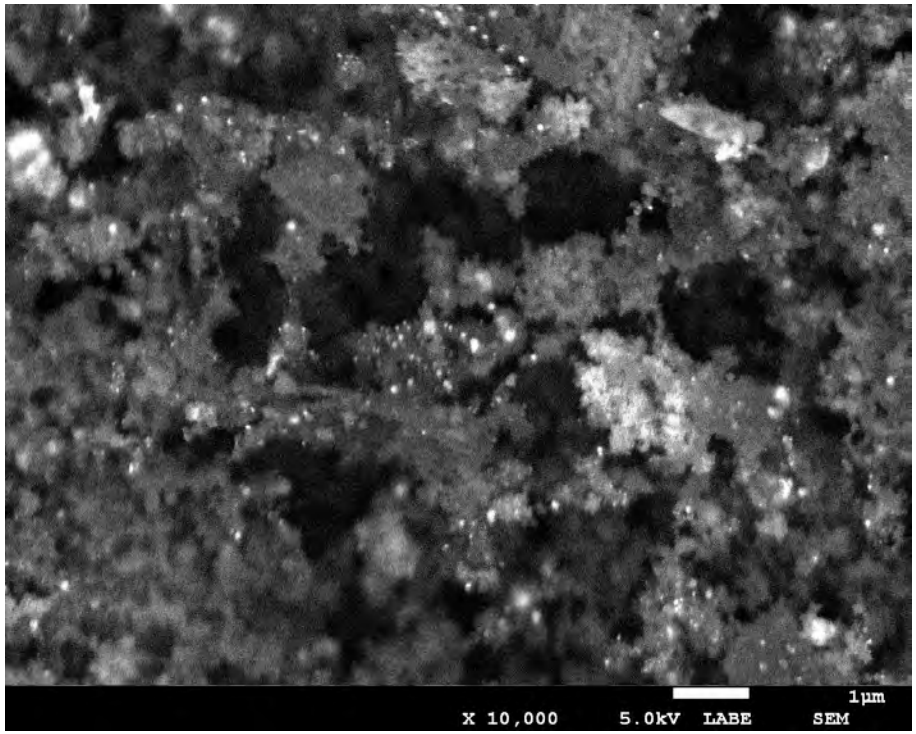
Material on surface consists of nano-sized agglomerates

Located 9 feet down range – 9 O'clock

SEM Stub 9

Backscatter SEM 5KX, 10KX



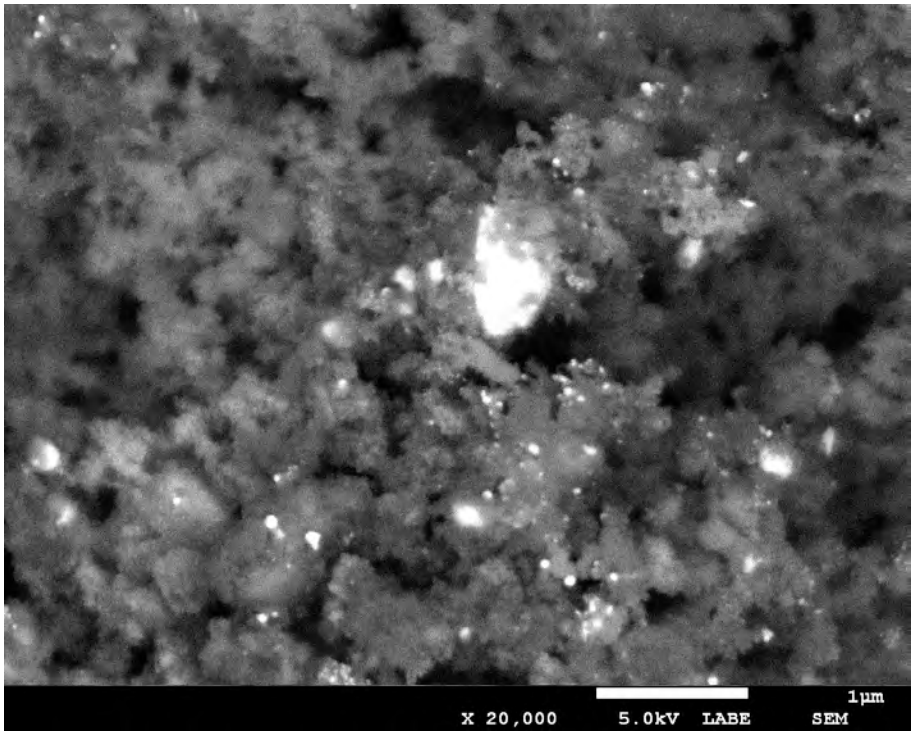


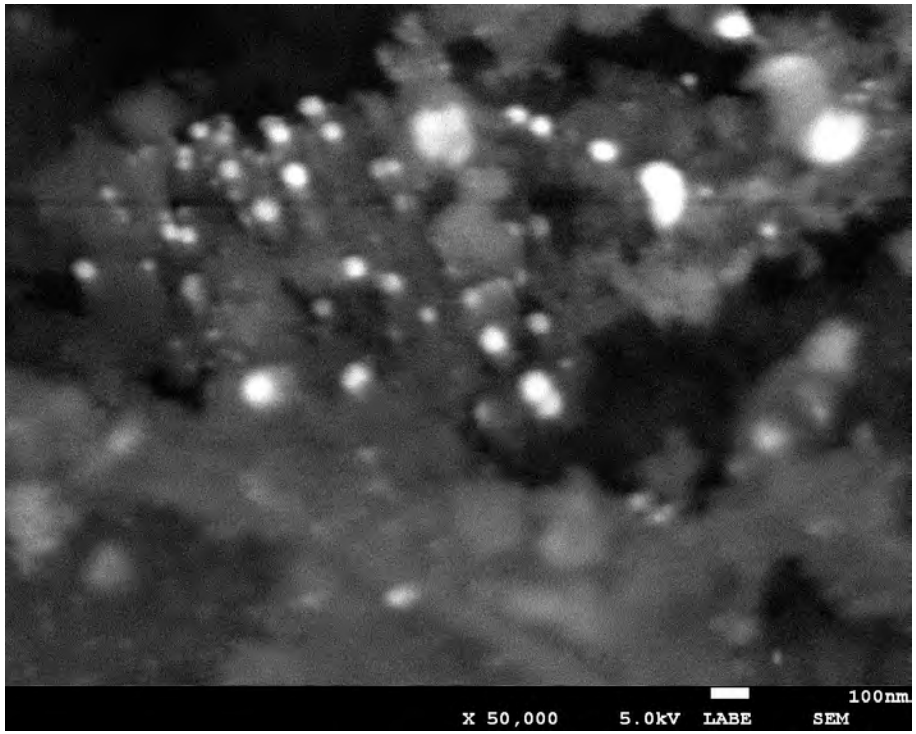
Nano-sized bright areas are high Z (Fe, Cu, Zn Ge, Al) mixed with darker carbonaceous debris.

Located 17 feet down range – 12 O'clock

SEM Stub 9

Backscatter SEM 20KX, 50KX



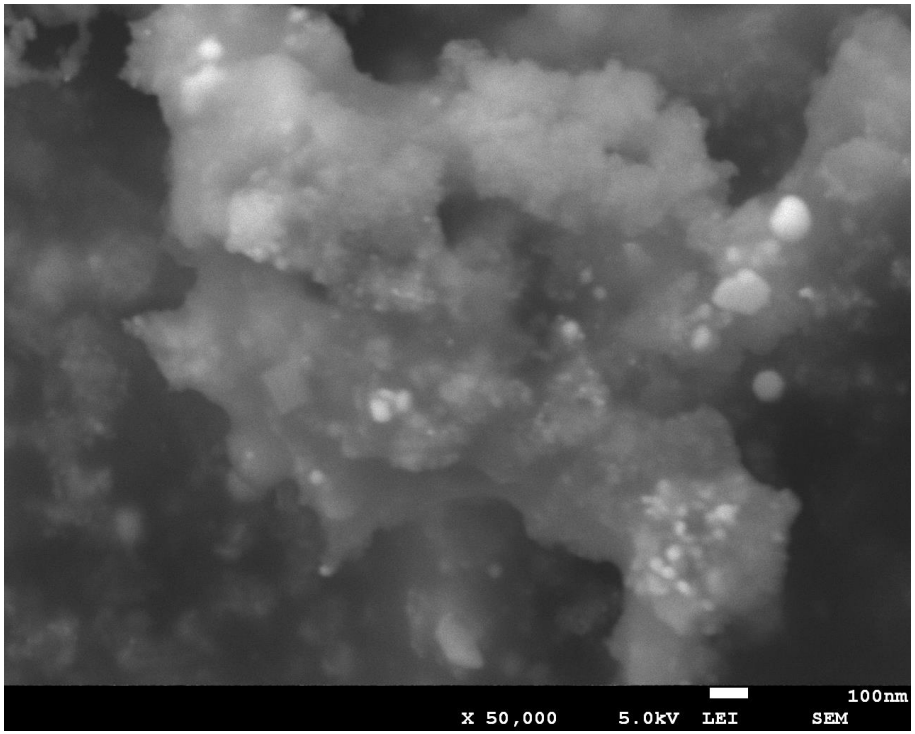


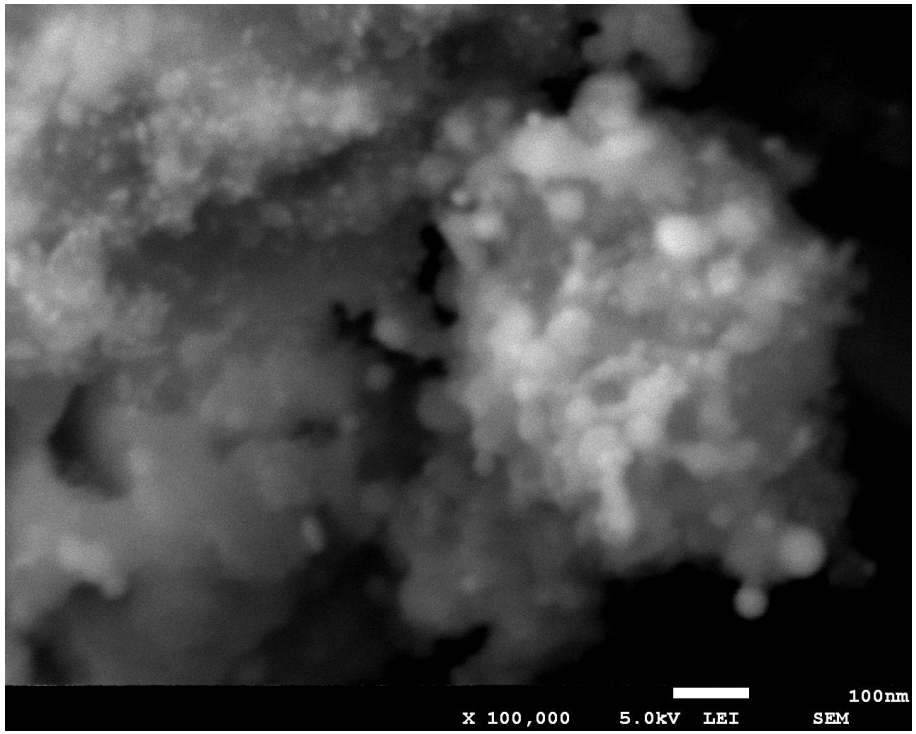
Nano bright areas appear to be solidified molten droplets of high Z material (Fe, Cu, Zn Ge, Al) mixed and covered with carbonaceous debris.

Located 17 feet down range – 12 O'clock

SEM Stub 13

Secondary Electron SEM (50KX, 100KX)



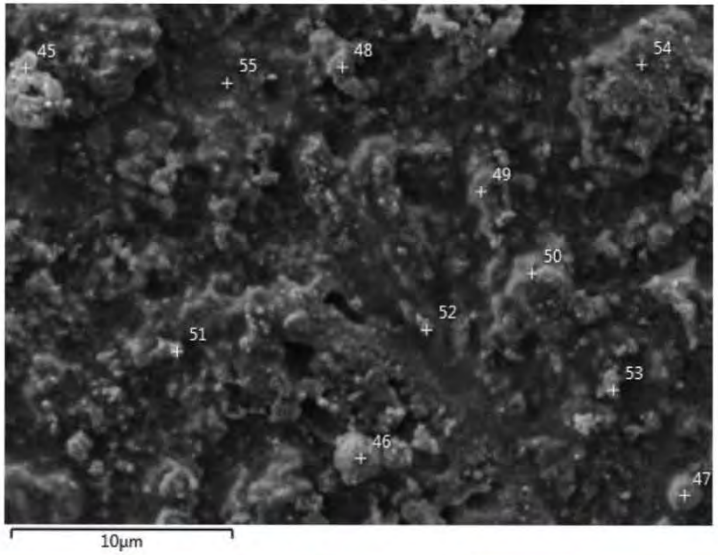


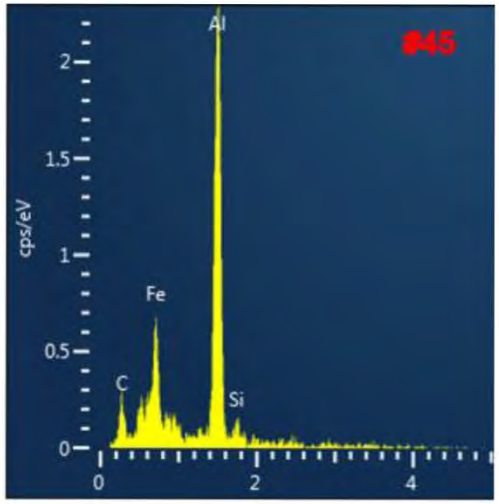
Note nano-scale particulates

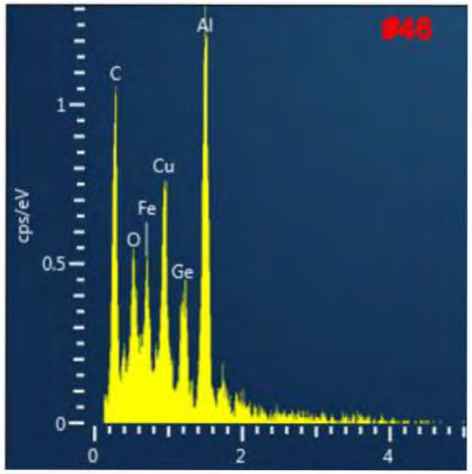
Located 11 feet down range – 3 O'clock

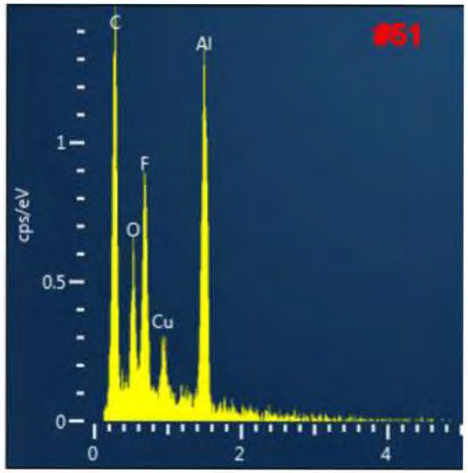
SEM Stub 13

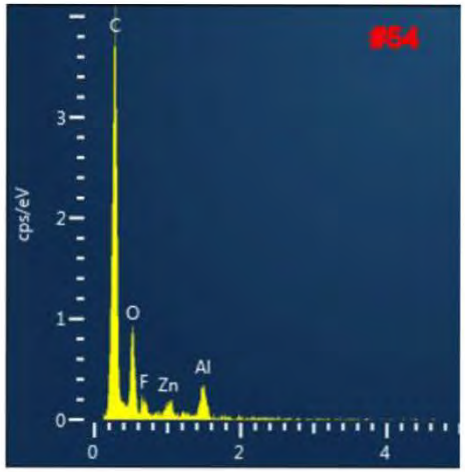
SEM EDS







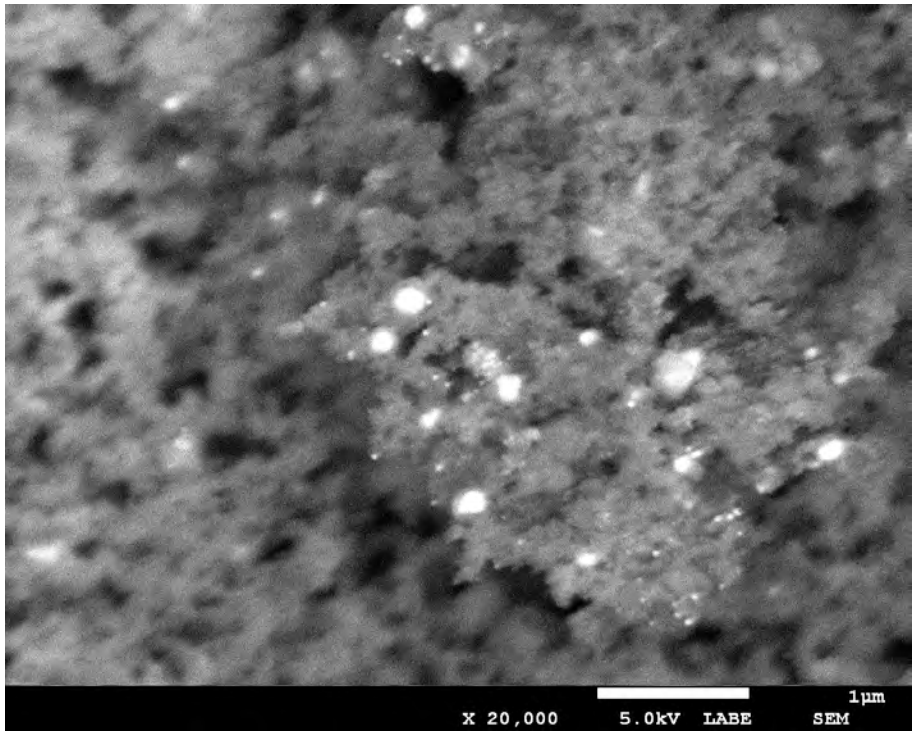


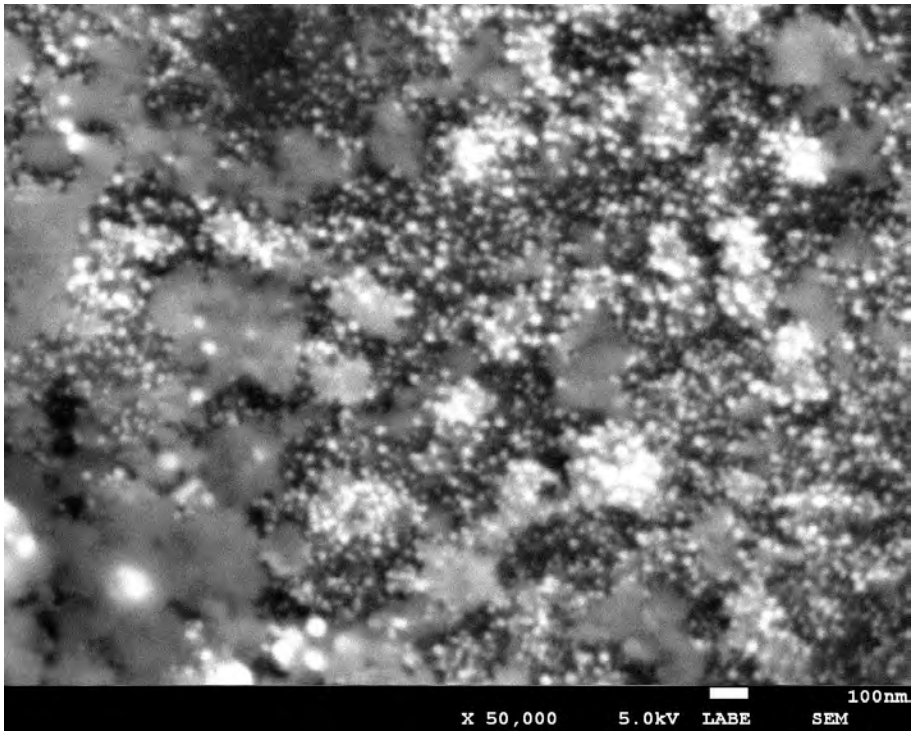


Al, C, F, Cu, O, Fe, and Ge are common.
Bright nano droplets contain Fe, Cu, Ge.

SEM Stub 14

Backscatter SEM 20KX, 50KX





Majority of the deposit is an agglomeration of nano carbonaceous material

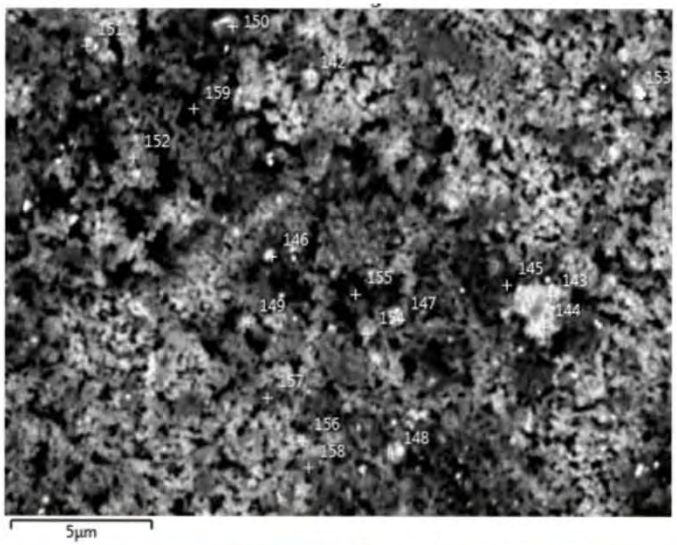
Note higher Z nano particles

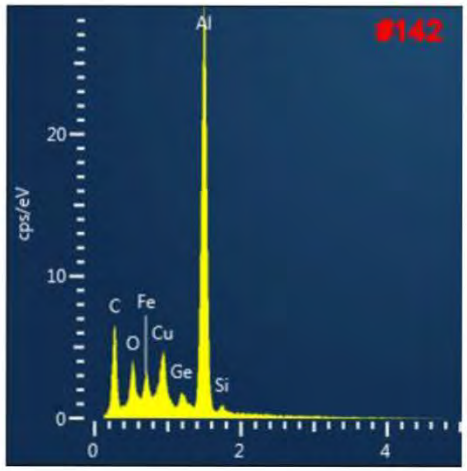
Located 15 feet down range – 3 O'clock

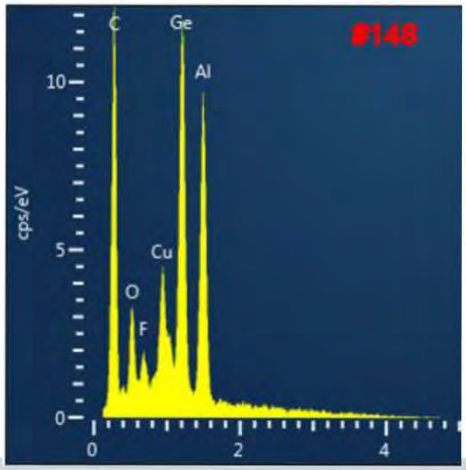
SBU Marking

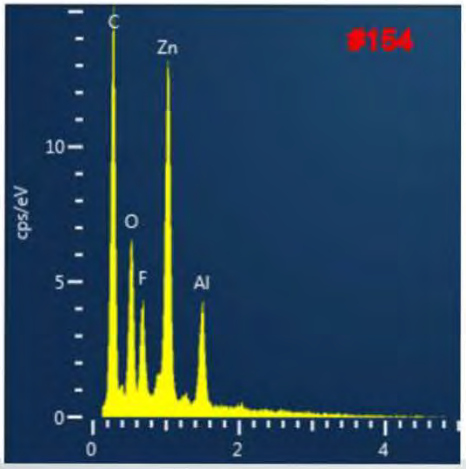
SEM Stub 14

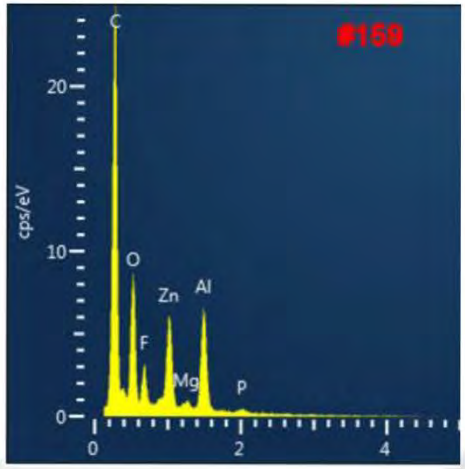
SEM EDS (5 KV)







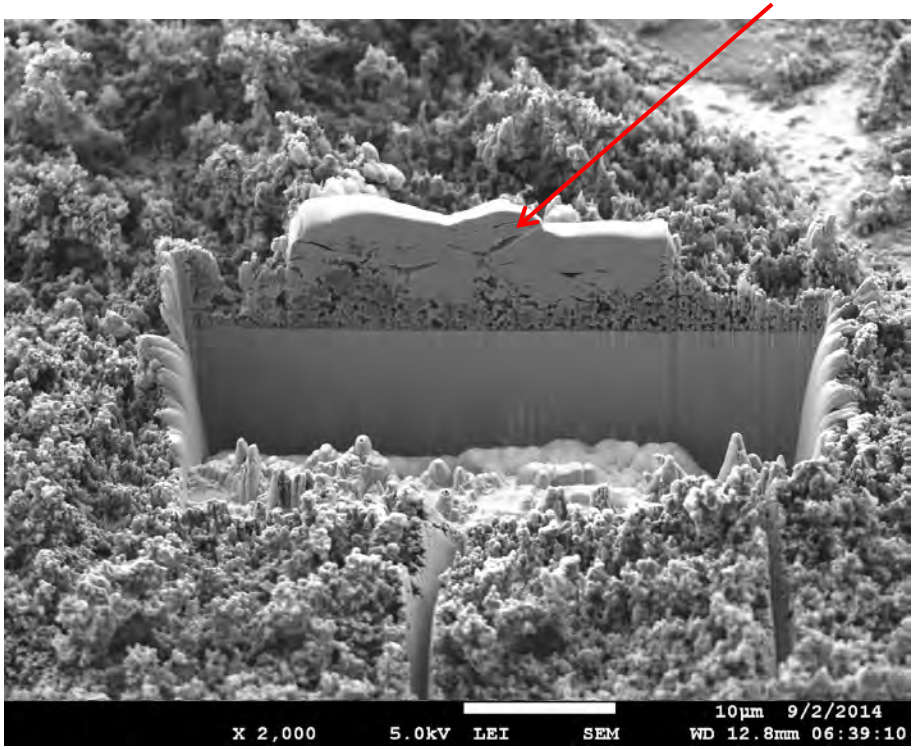


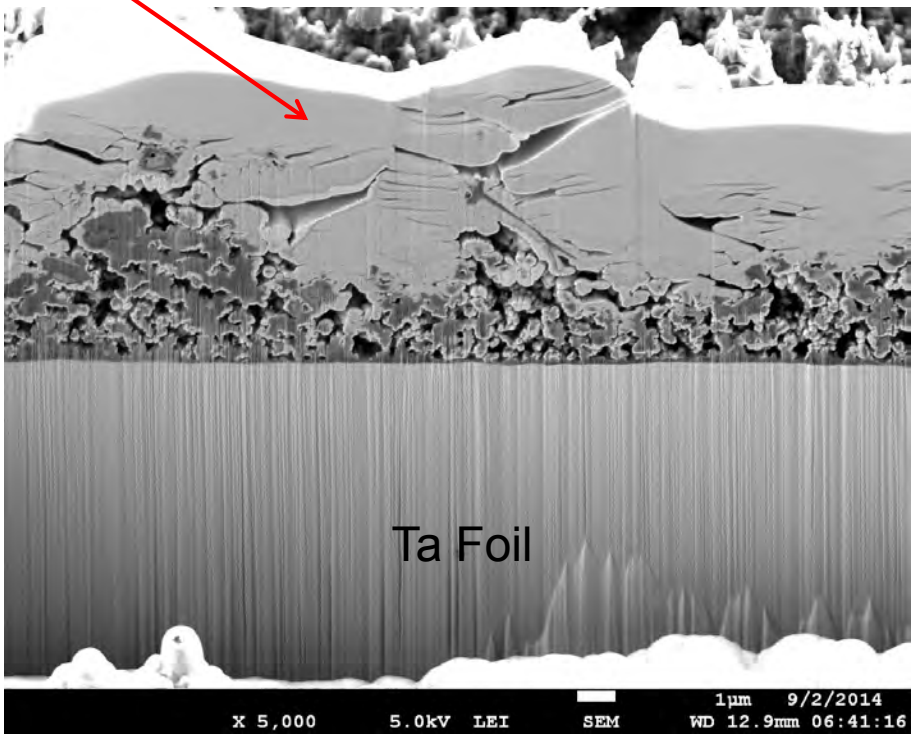


C is ubiquitous - Al, Zn, Ge, F and Cu are common.

SEM Stub 14: Focused Ion Beam (FIB) Cross Section

Secondary Electron SEM 2KX, 5 KX



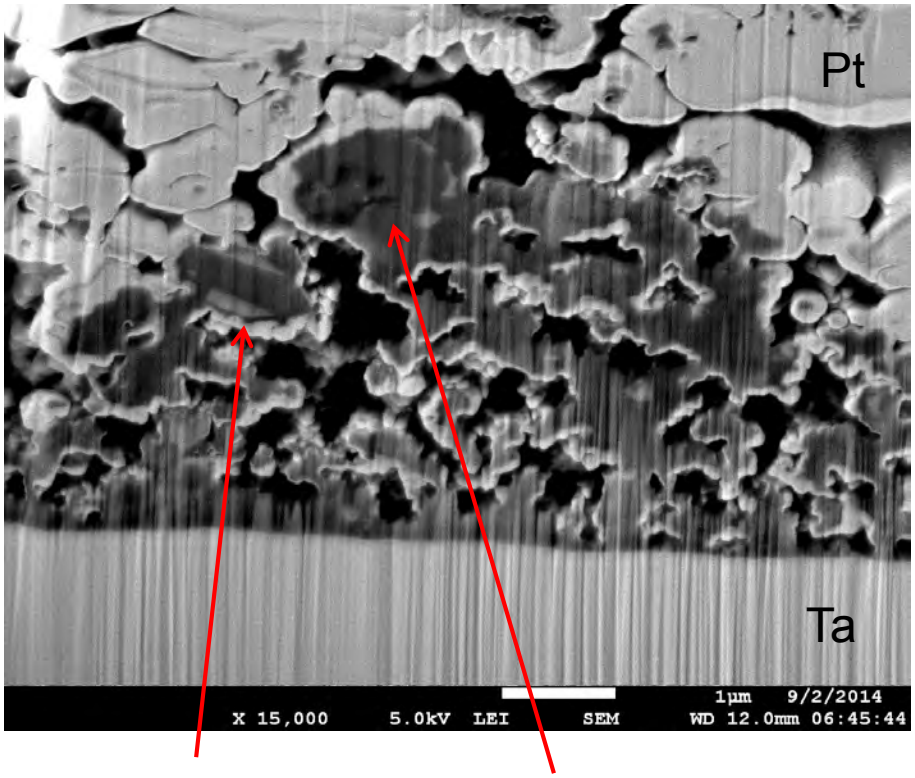


45 degree tilt

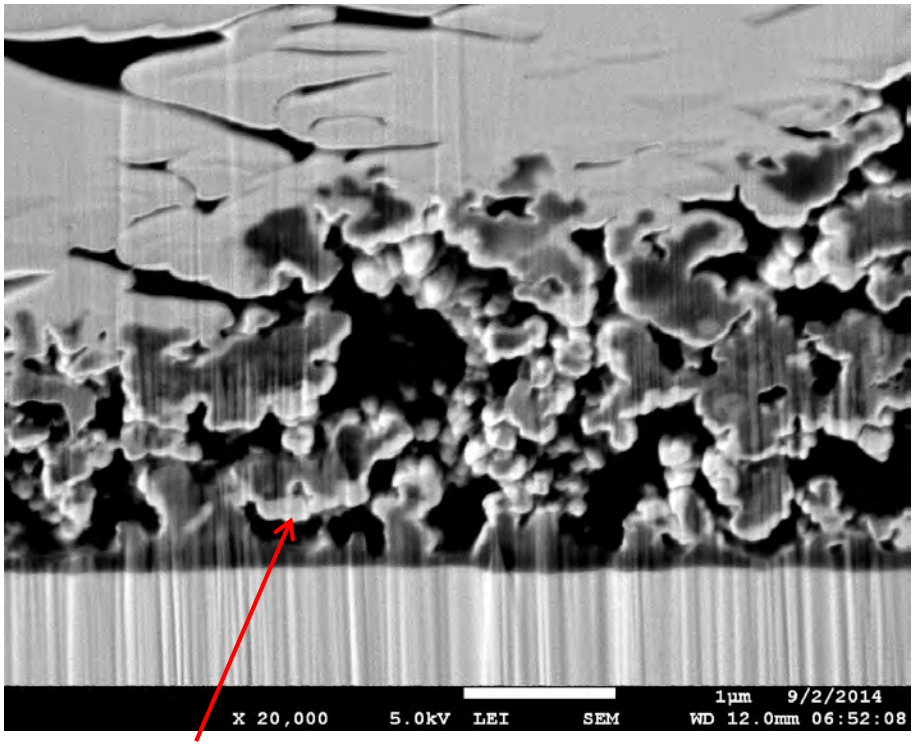
Deposit is several microns thick consisting of loose debris which is composed of agglomerates of nano particles.

SEM Stub 14: FIB Cross Section

Backscatter Electron SEM 15KX, 20 KX



Higher Z fragments in carbonaceous matrix

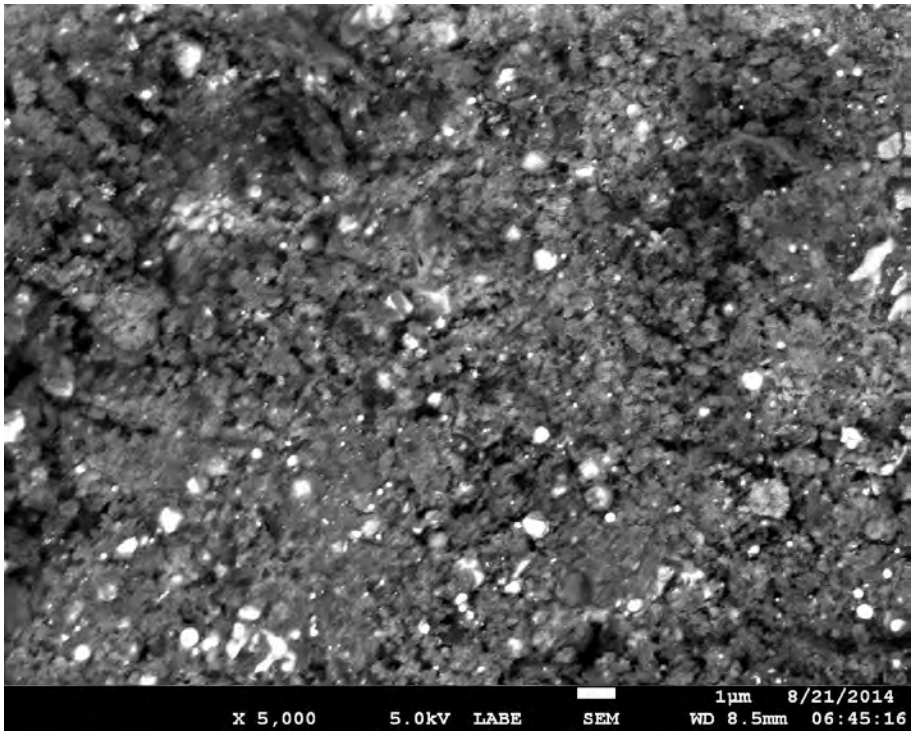


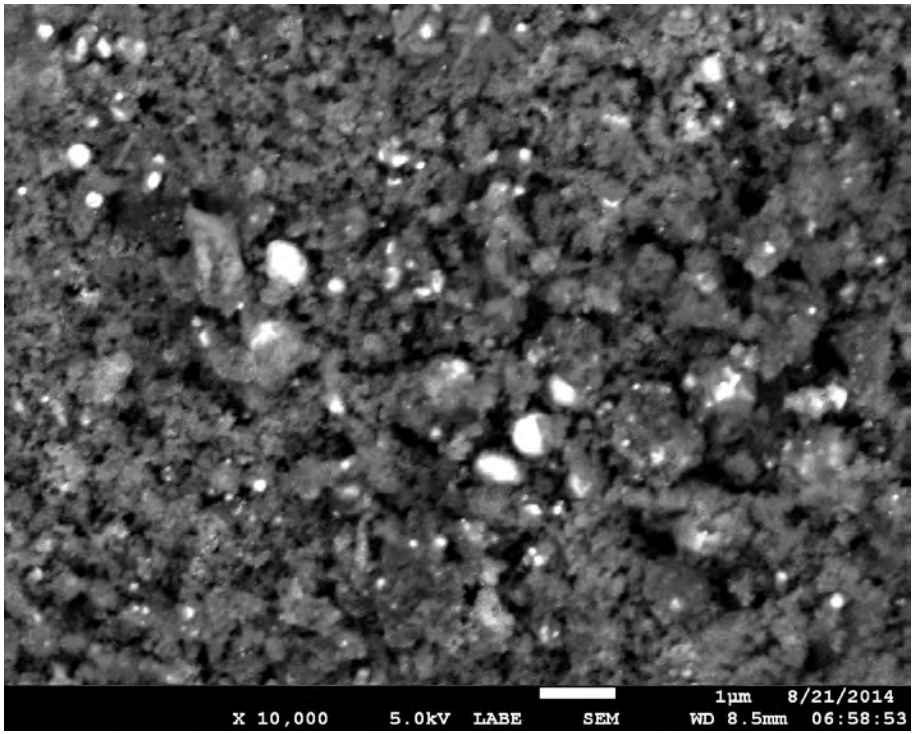
Light rind on particles is probably Ta ejected into cavities during the trenching operation or infusion of Pt.

EDS analysis was hampered by relatively large sampling volume and introduction of significant Pt, Ta and Ga. C, O and some Al detected.

SEM Stub 20

Secondary Electron SEM 5KX, 10KX





Material on surface consists of nano-sized agglomerates.
Darker areas are carbon rich. Bright spots contain Fe, Cu, Zn, Ge and represent solidified molten droplets partially covered with carbonaceous nano particles.

Located on down range back wall.

Summary of Area EDS Analyses

v	Stub #4	Stub #13	Stub #14	Stub #9	Stub #20
C	57.7	78.0	61.9	61.1	61.7
O	20.1	11.9	16.1	18.8	16.2
F	7.4	1.8	4.4	5.7	2.9
Na	0.90	0	0	0.35	0
Mg	0.27	0.12	0.38	0.36	0.39
Al	5.4	3.6	5.8	8.7	12.5
Si	1.1	0.53	0	0.88	0.45
P	0	0.11	0.30	0.28	0.35
S	0	0	0	0.03	0
Cl	0.13	0.09	0.12	0.10	0
Cr	0	0	0.03	0.16	0.38
Fe	0.28	0.22	0.28	0.64	1.42
Cu	0	0.09	0.14	0.25	1.27
Zn	3.4	0.16	7.7	1.9	2.0
Ge	0.07	0	0	0.18	0.40
Ta	3.4	3.4	2.9	0.40	0
Distance Down Range	9 ft	11 ft	15 ft	17 ft	19 ft
Azimuth*	9 O'clock	3 O'clock	3 O'clock	12 O'clock	End Panel

All values atomic %

Ta is from the underlying Ta sheet

* Facing down range

Averages of (2) 12 mm^2 areas

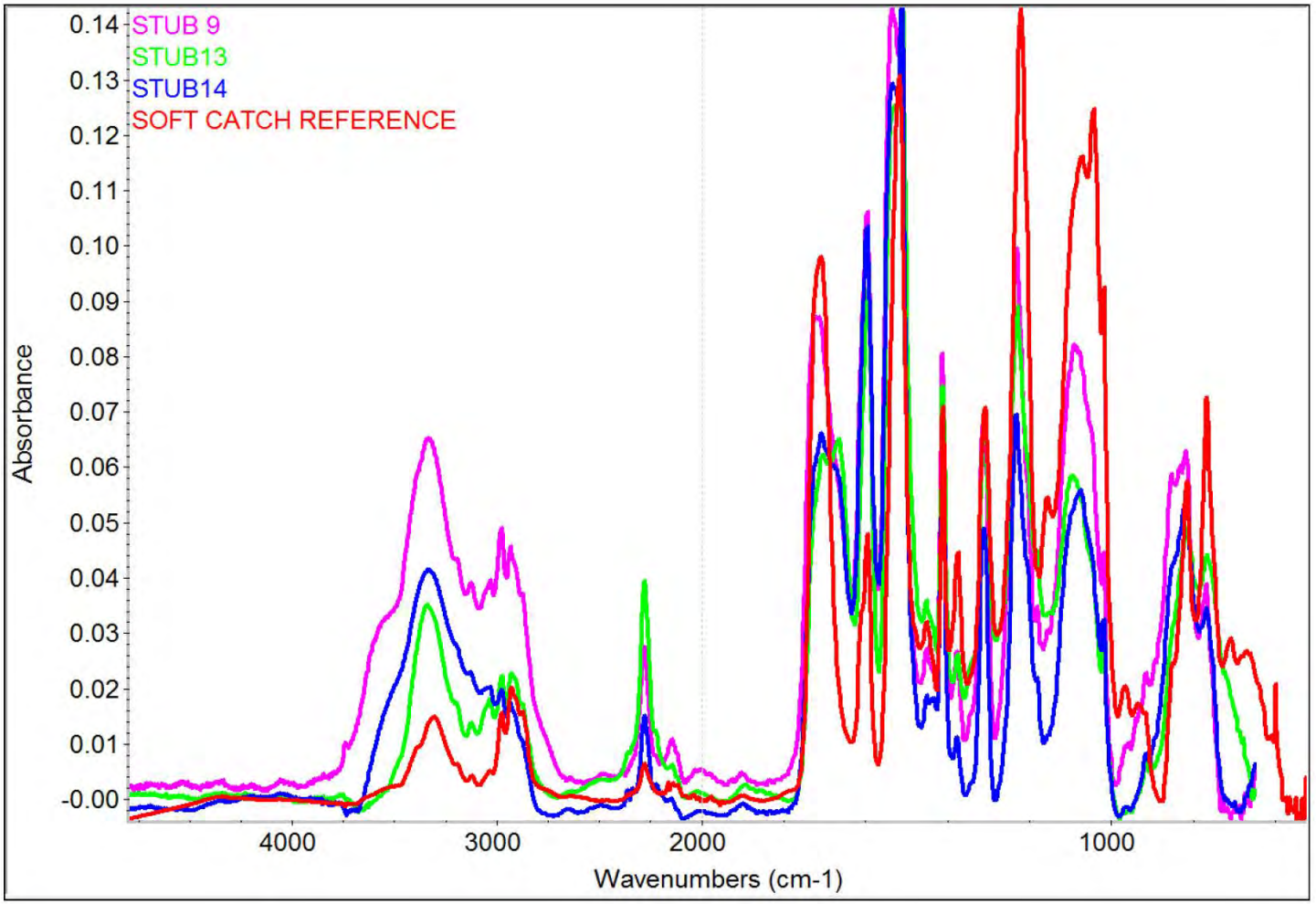
No consistent trends in elemental distribution.

Summary of Area EDS Analyses

- The majority of the deposits consist of carbon and oxygen (77- 89 at%).
  - Primarily from pyrolyzed soft catch (based on Debris-LV results).
  - Some contribution form MLI and C-C composite face sheet.
- Fluorine (1.8 - 7.4 at%) is from Teflon wire insulation and?
- Metal nano particles are common
  - Aluminum (3.6 - 12.5 at%) is from the aluminum honeycomb panels, structural core, nadir-zenith panels and COPV tank.
  - Zinc (0.2 - 7.7 at%) is from an unidentified source.
  - Iron (0.2 - 1.4 at%) is from stainless steel tubing and solenoids.
  - Copper (0.09 - 1.3 at%) is from wiring and solenoids.
  - Germanium (0.07 – 0.40 at%) is from the solar cells.

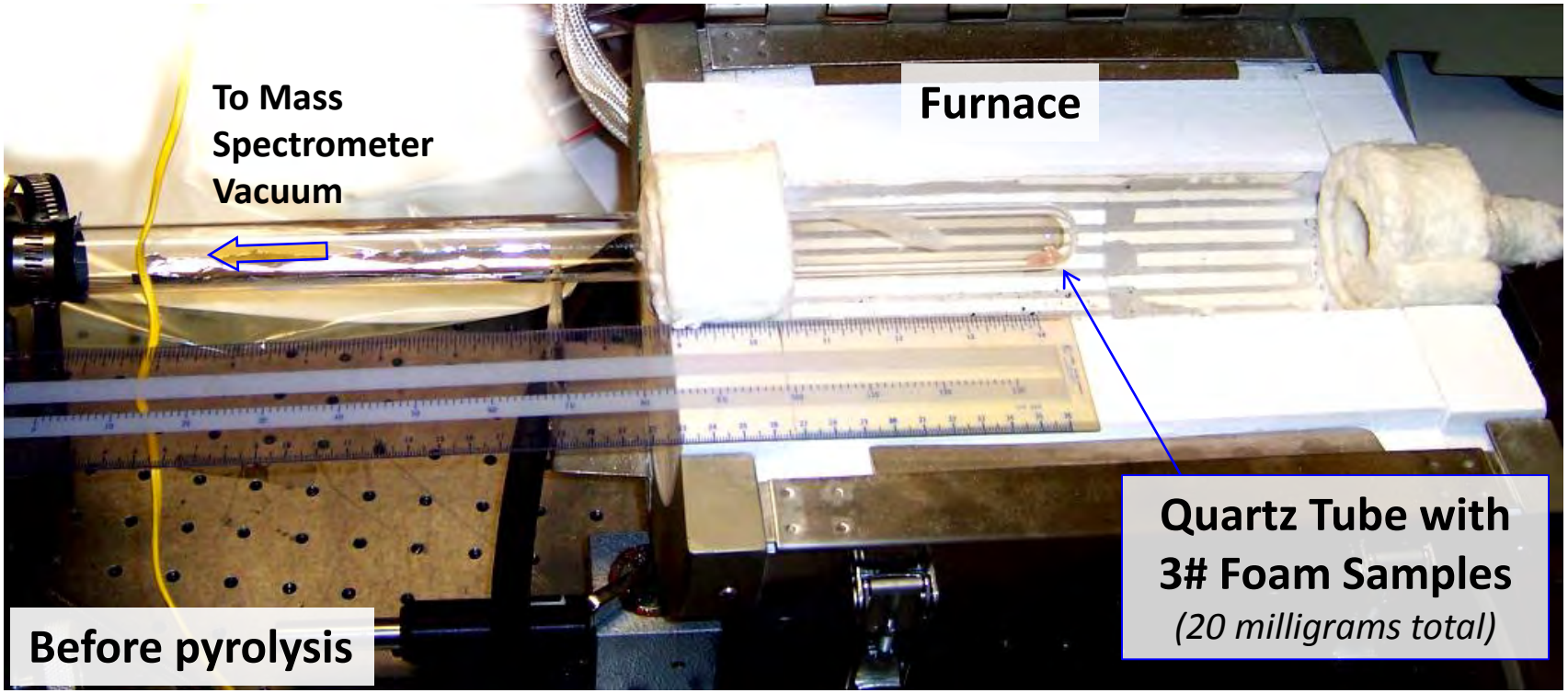
Post Test: SEM Stubs

FTIR – Qualitative Diffuse Reflectance



Significant soft catch signature is present on SEM stubs. Some, but not all, is in the form of soft catch foam fragments which give samples a sparkly appearance.

Laboratory Foam Pyrolysis Experiment

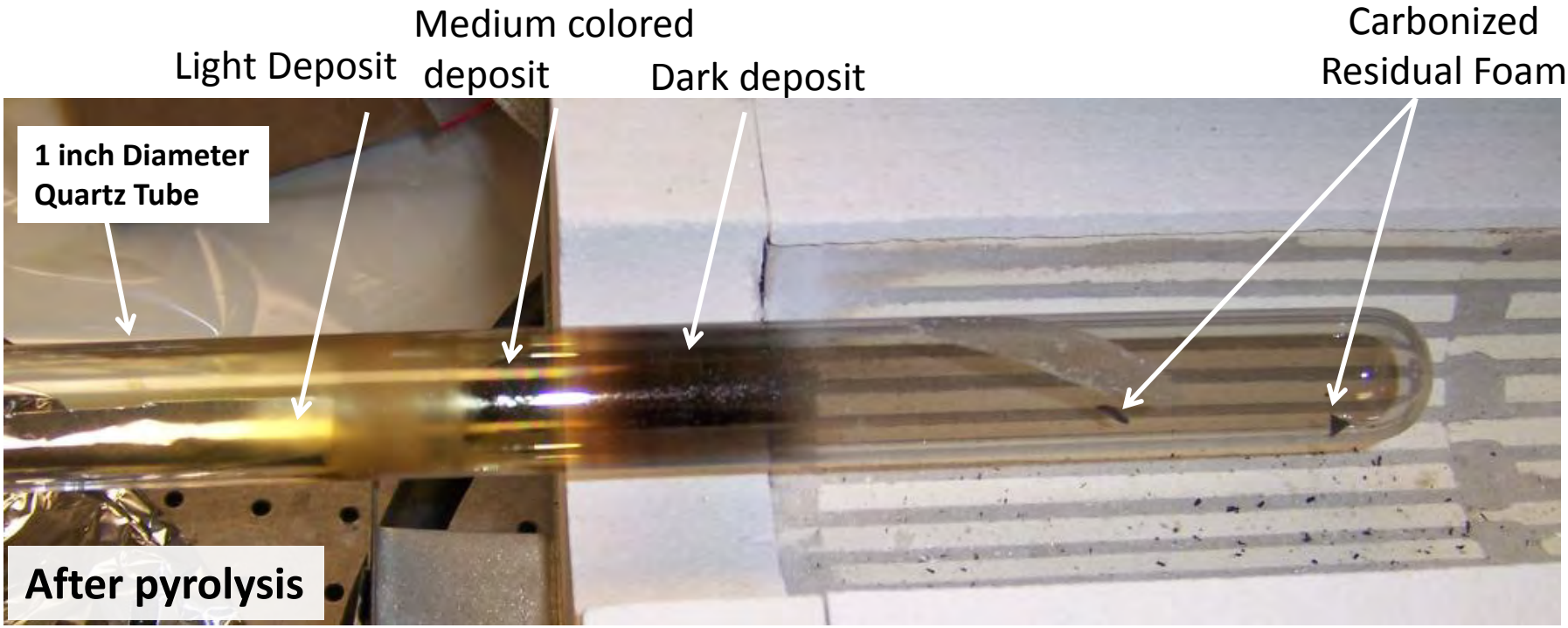


~ 0.001 Torr Throughout Pyrolysis => λ ~ 10 inches

30 milligram pieces of 3# foam pyrolyzed in a quartz tube under vacuum in order to simulate exposure to plasma from hypervelocity impact.

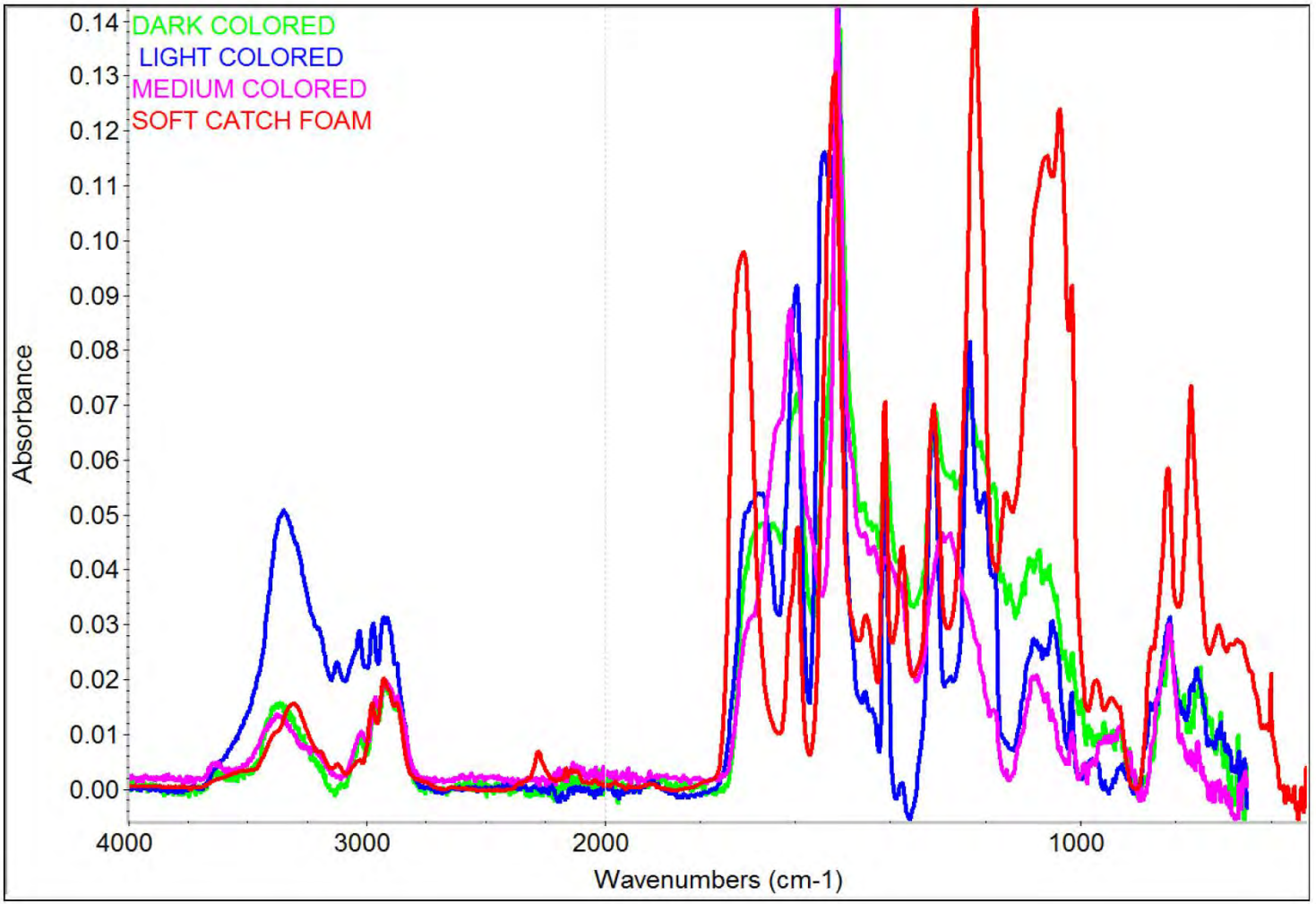
Condensate residues in cool portion of tube outside the furnace were analyzed by FTIR .

Laboratory Foam Pyrolysis Experiment



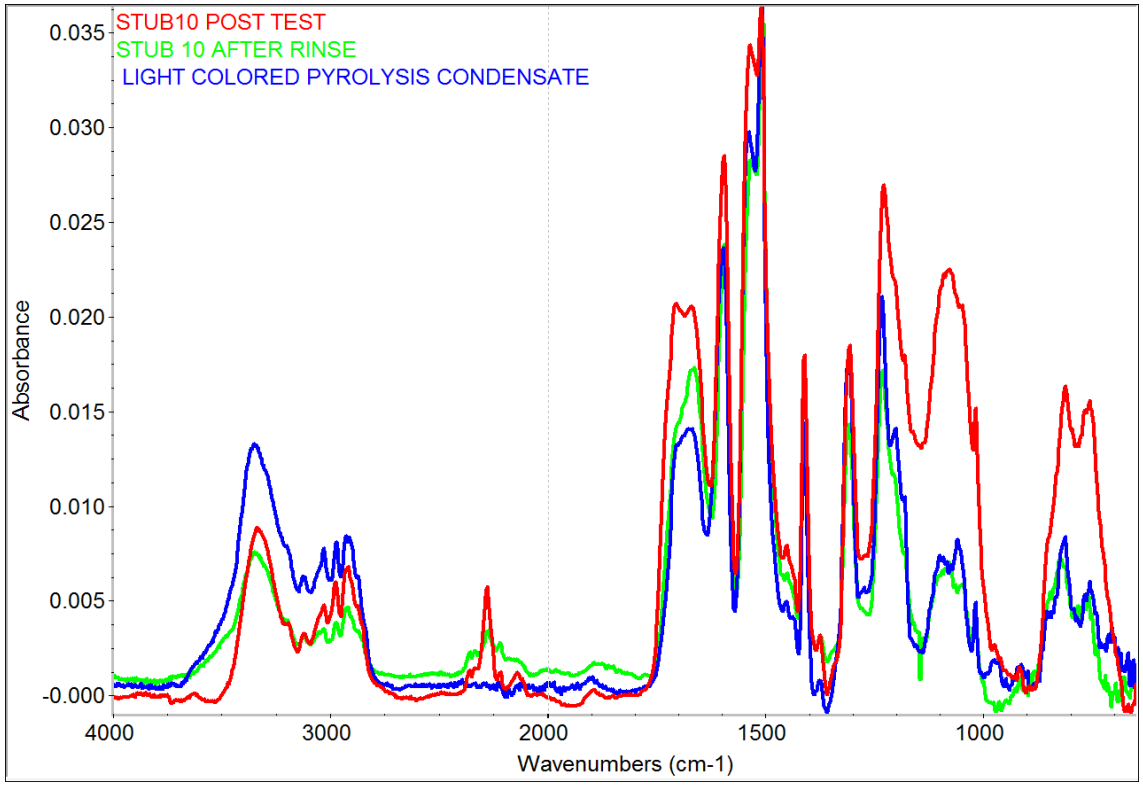
SBU Marking

Condensate Removed from Tube



Various colors of condensate have similar but slightly different spectra that resemble soft catch foam.

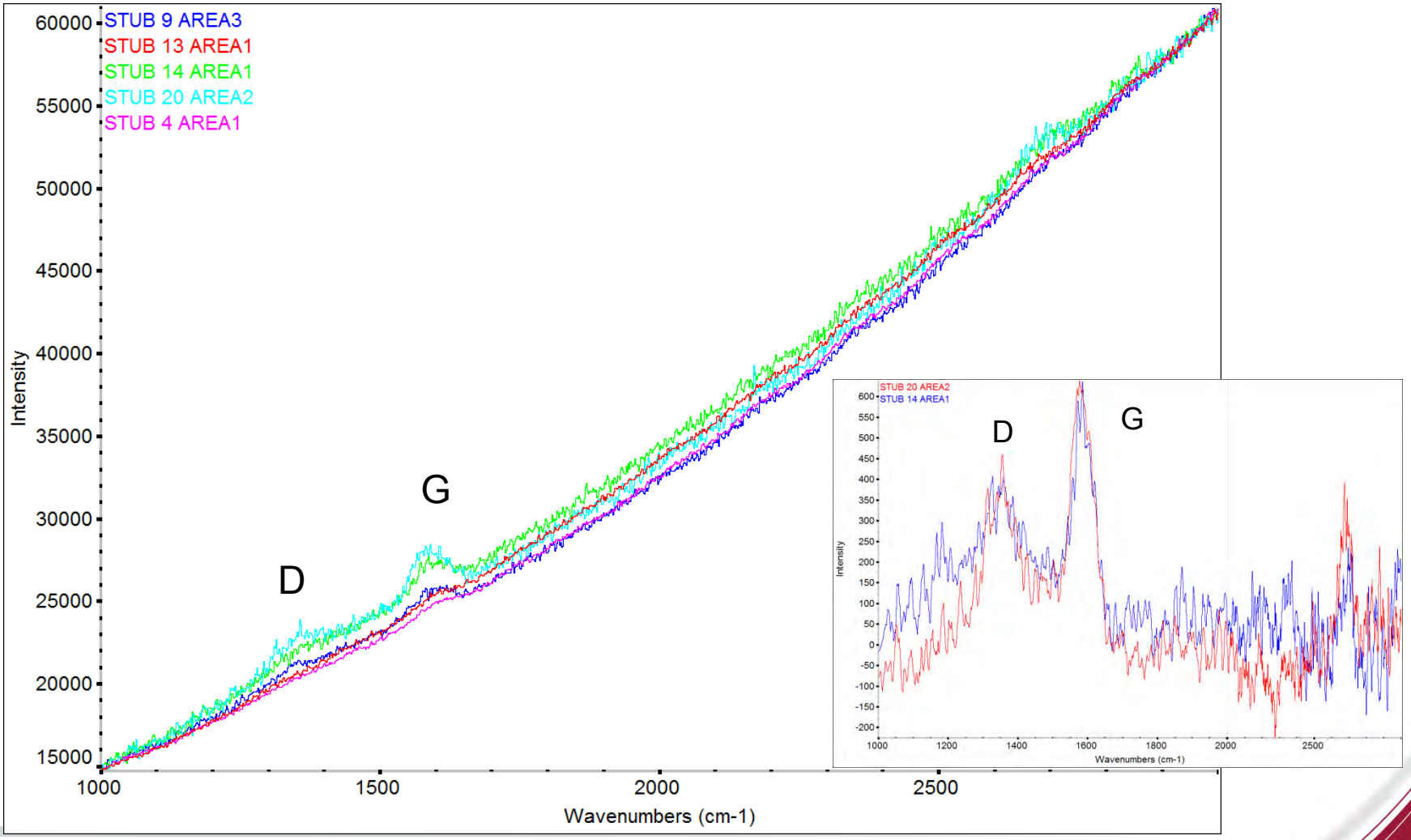
Debris-LV Stub 10 vs. Pyrolysis Condensate



- Spectrum of soft catch condensate is similar to that on SEM stubs, before and after rinsing stubs with isopropyl alcohol (IPA). Alcohol rinse only removed loose fragments.
- SEM stubs are covered with a thin layer of soft catch condensate in addition to fragments. Probably a result of close proximity to soft catch panels exposed to plasma.

Since DebriSat also used soft catch - SEM stubs are probably also covered with a thin layer of soft catch vapor condensate. DebriSat stubs were not rinsed with IPA.

SEM Stubs: Raman Spectroscopy



Disordered (D) and crystalline graphite (G) bands observed in some areas.
Graphitic bands also seen in Debris-LV deposits.

Witness Plate: Post Test

Witness Plate Samples:

<u>Direct Exposure</u>

(4) 1” fused silica **(D)**
(1) 1” Z-93 painted Al
(1) 1” Aluminum
Multi layer insulation

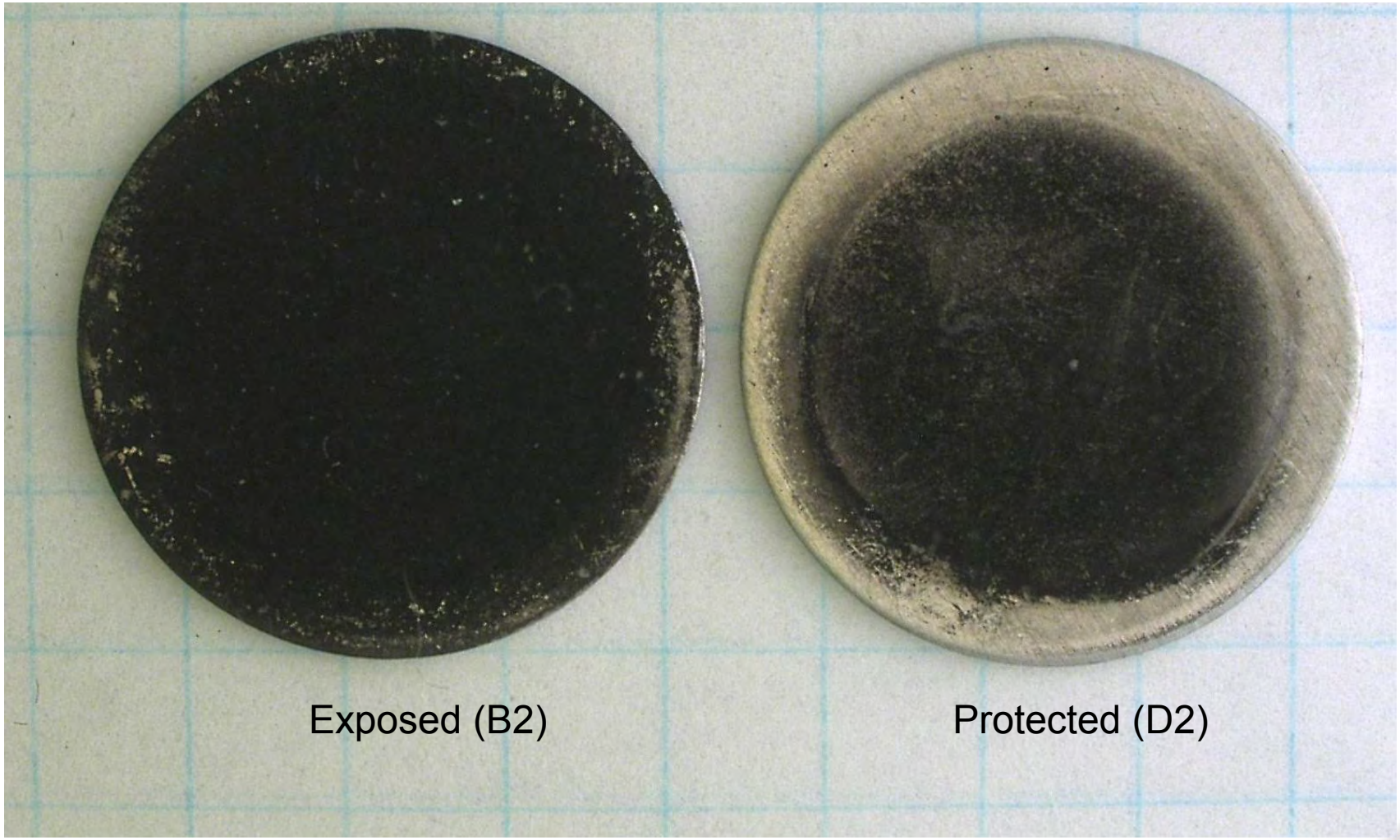
<u>Protected Under Whipple Plates</u>

(2) 1” fused silica
(1) 1” Z-93 painted Al
(1) 1” Aluminum
(1) 1” NaCl (cleaved)
(1) Cu sheet
Ge ATR crystal **(D)**
Solar cell

(D) = destroyed

Top Whipple plate received a significant impact. Assembly is completely covered with black soot. Many witness plate samples were fractured/destroyed.

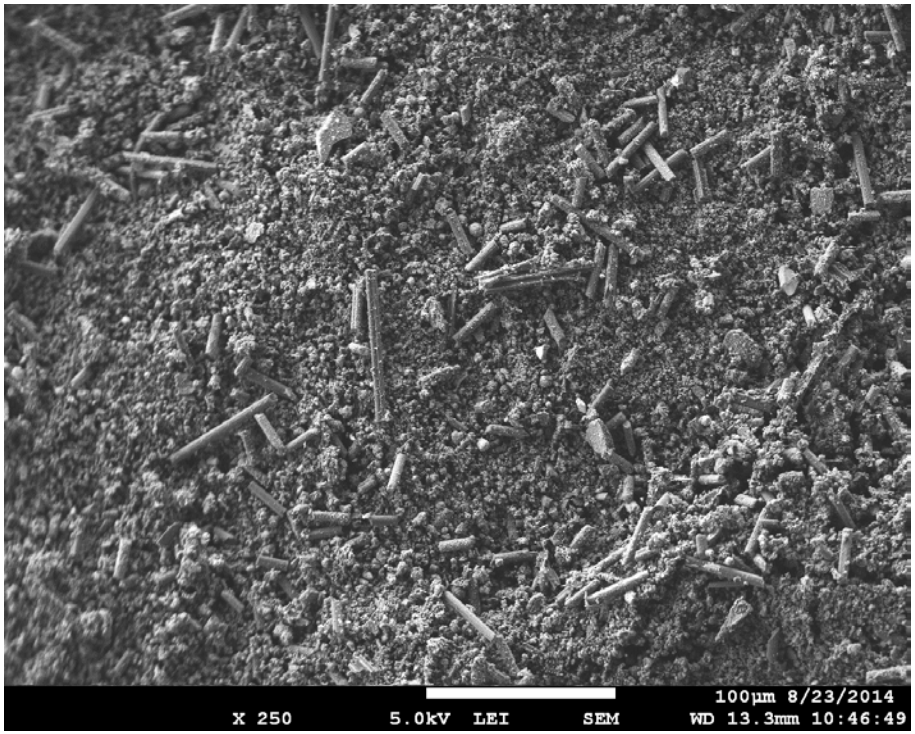
Witness Plate Post Test : Aluminum Disks

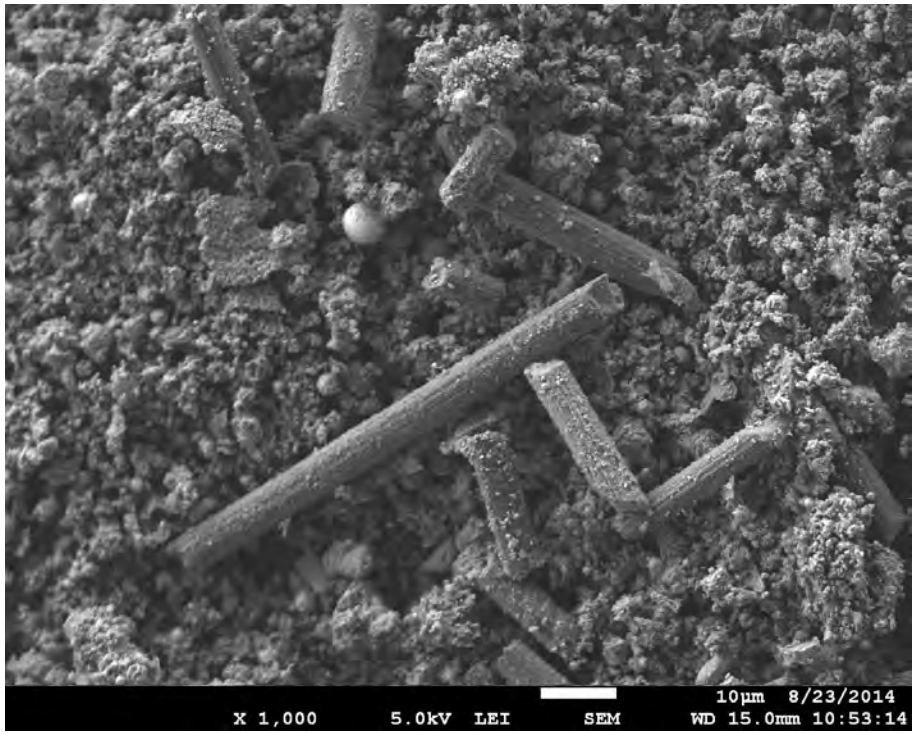


Significant accumulation of debris, especially on exposed disk

Witness Plate Post Test : Aluminum Disk B2 (exposed)

Secondary Electron SEM Images (250X, 1KX)

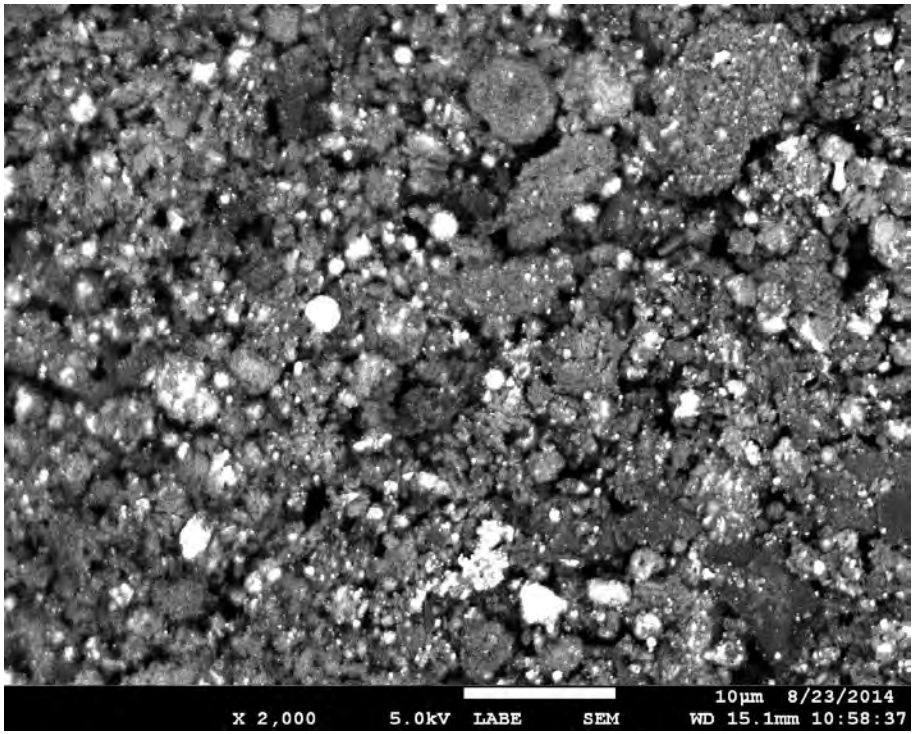


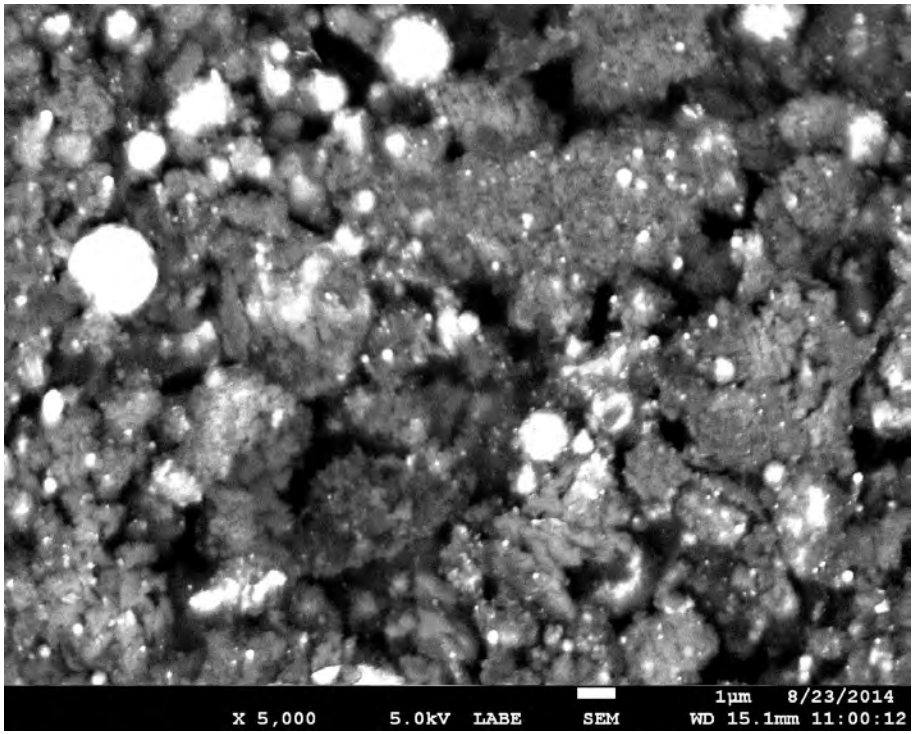


Note fragments of carbon fibers and round solidified droplets. Carbon fibers are from the composite face sheets on the honeycomb structural panels.

Witness Plate Post Test : Aluminum Disk B2 (exposed)

Backscatter Electron SEM Images (2KX, 5KX)

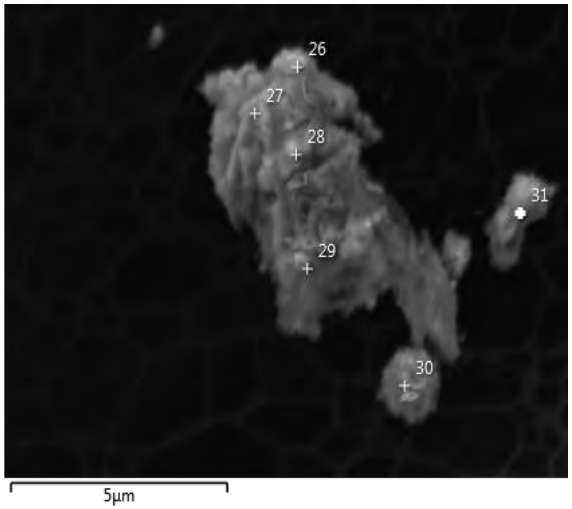


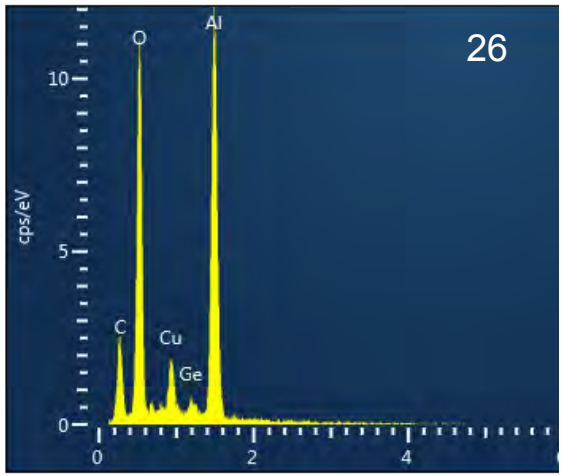


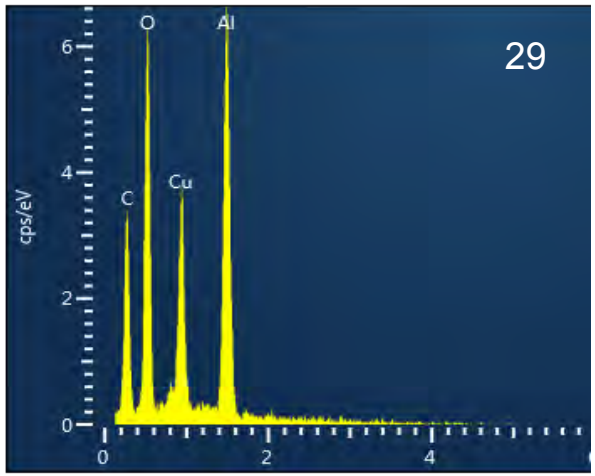
Round solidified droplets are higher Z (Al, Fe, Cu, Ge, Zn) and indicate melting of material as a result of impact.

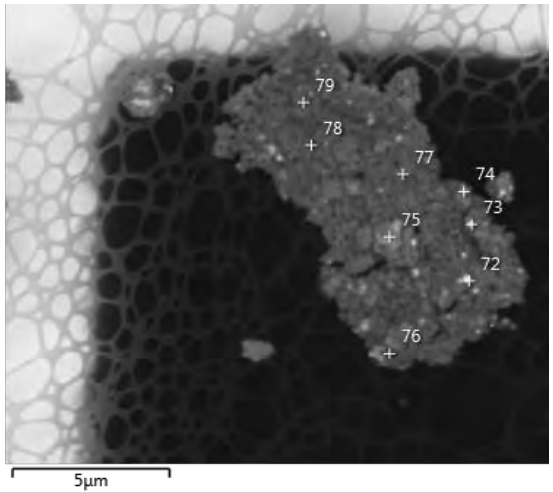
Witness Plate Post Test : Aluminum Disk B2 (exposed)

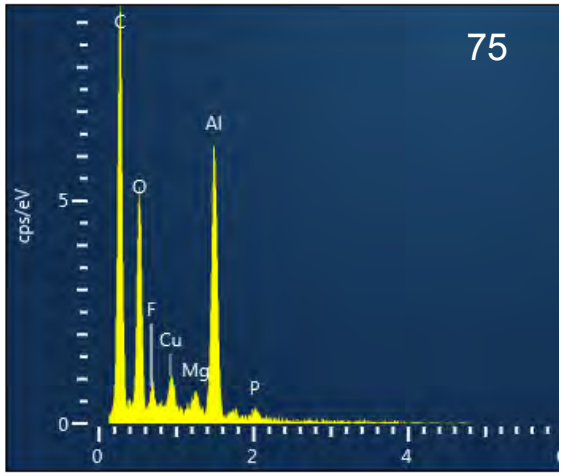
SEM Images of particles suspended on lacey carbon films on Cu TEM grid

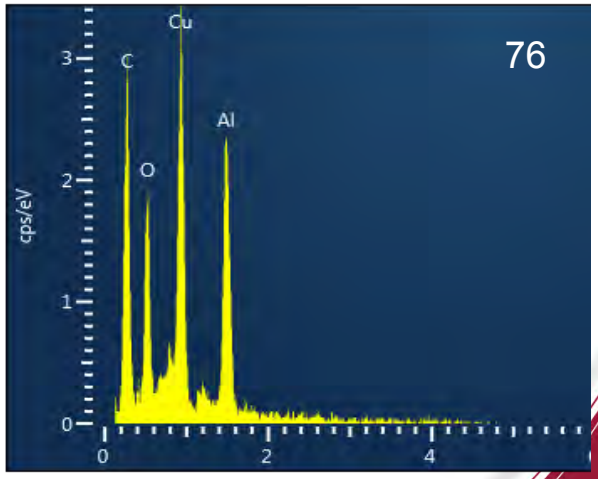






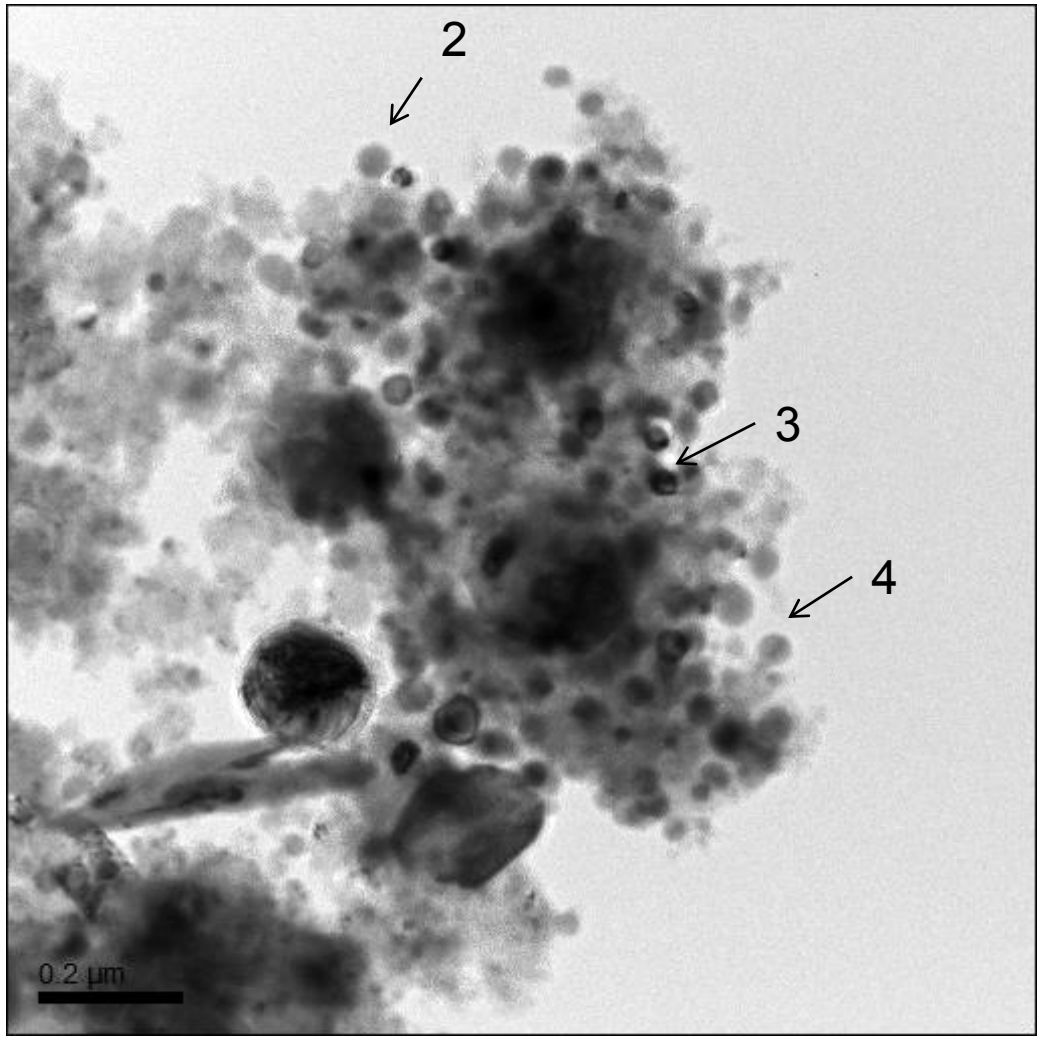






Particles are rich in C, O, Al and Cu. Minor C and Cu can be from grid but significant amounts are in the particles. Many particles appear to be aluminum oxide.

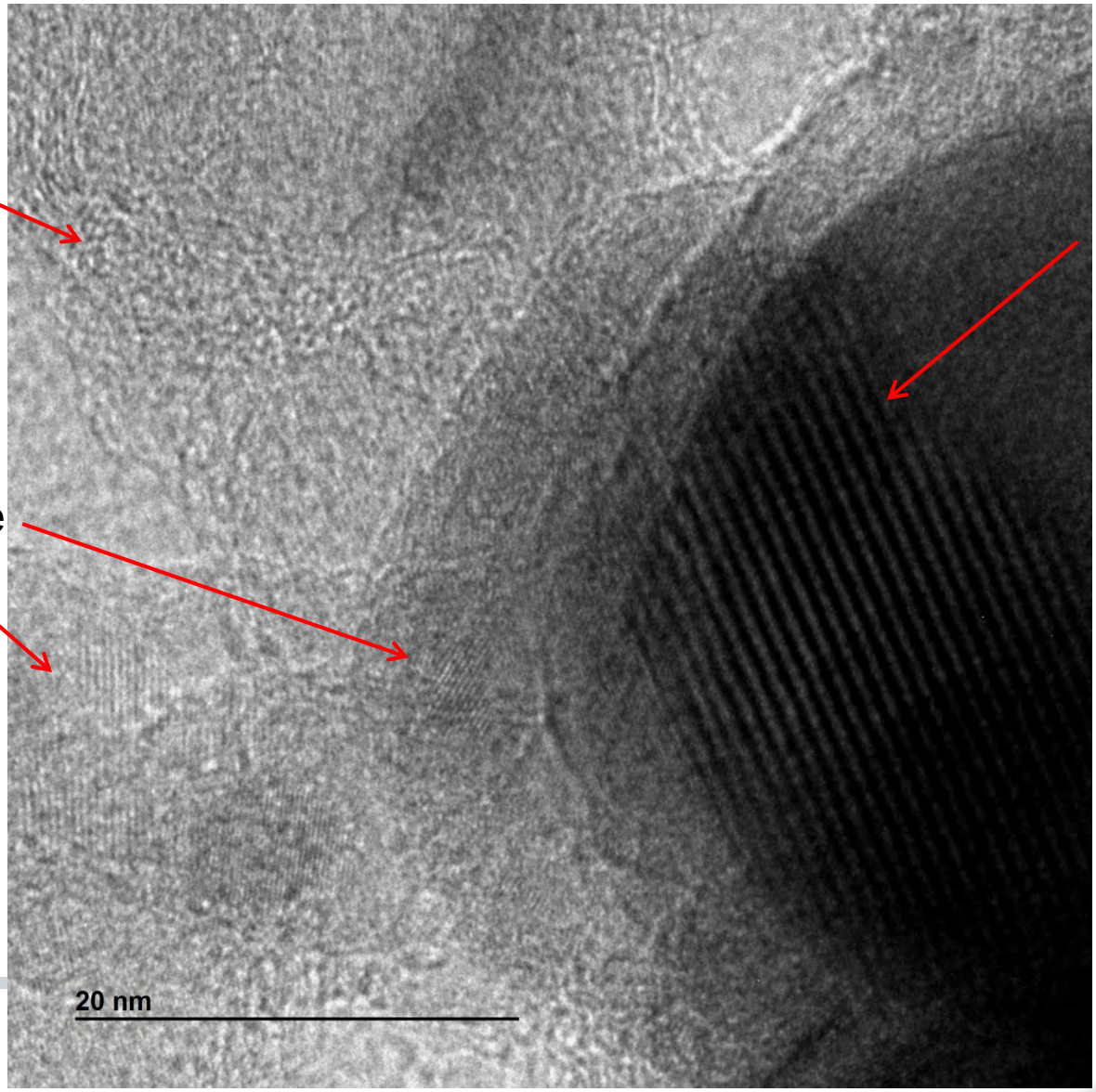
Witness Plate Post Test: Al Disk D2 Debris Transferred to TEM Grid:
Bright Field (BF) - TEM



Location 5

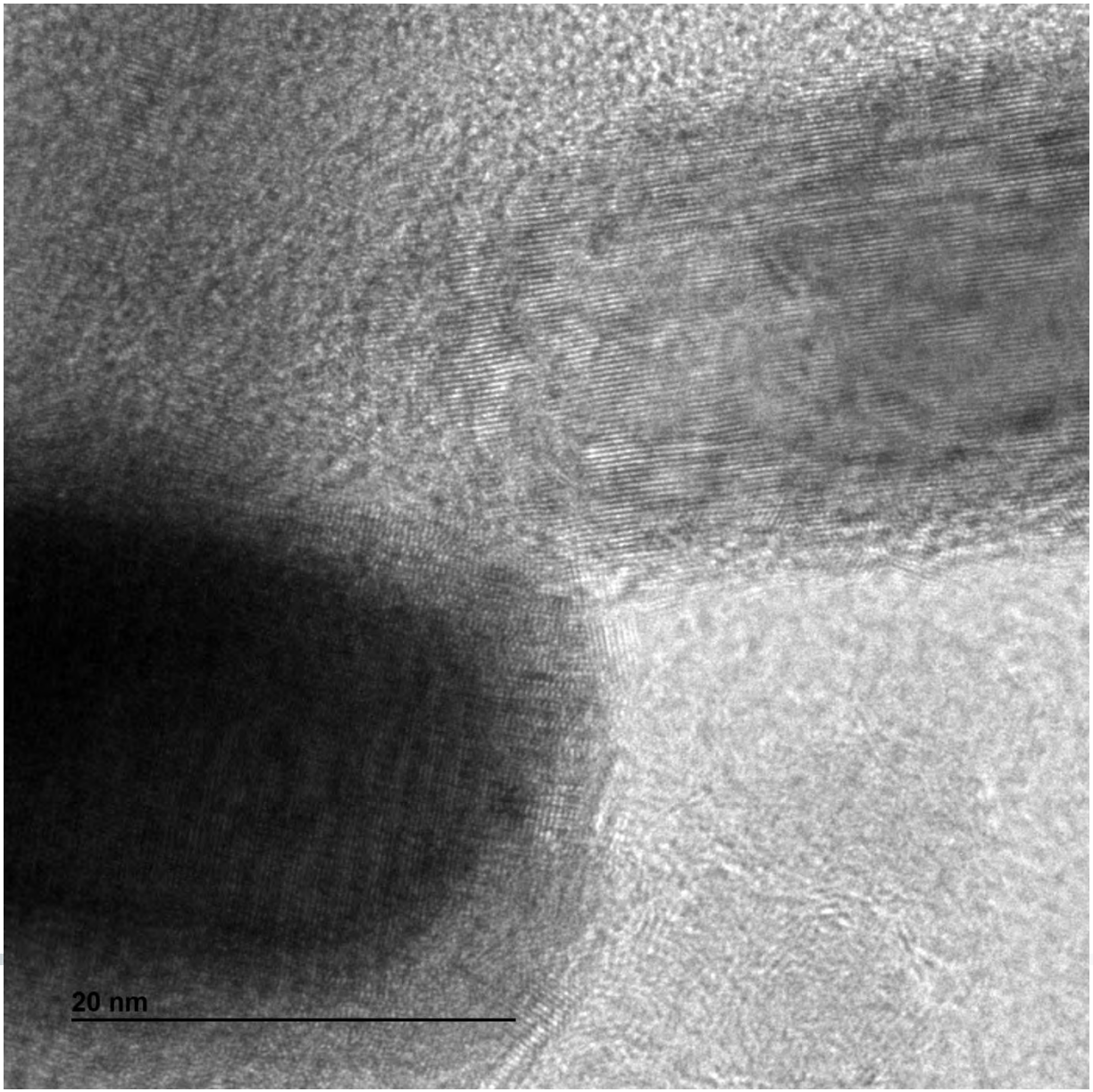
Material is an agglomeration of nano particles. Diffraction contrast in some particles indicates crystallinity.

Witness Plate Post Test D2: Area 3 BF-TEM



Crystal size is large in proportion to the size of the particles

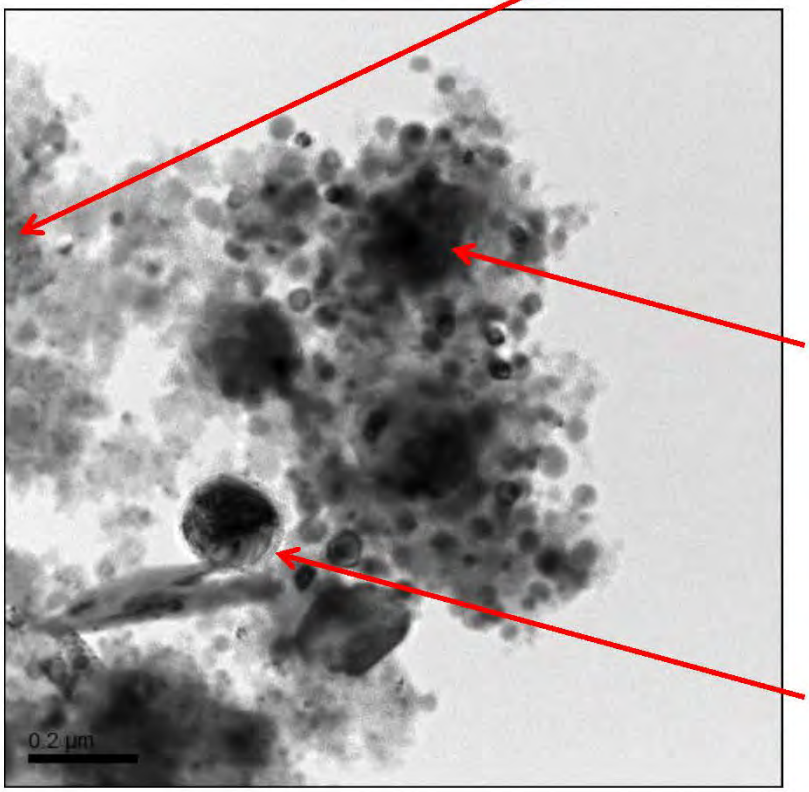
Witness Plate Al Disk D2 : Location 4 Area 6 BF-TEM

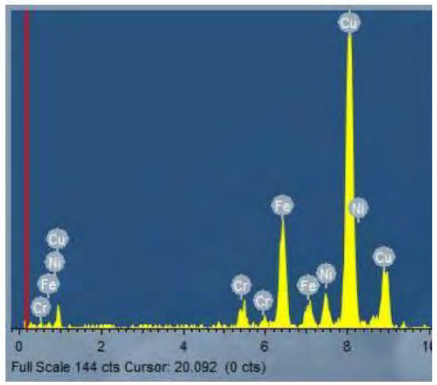


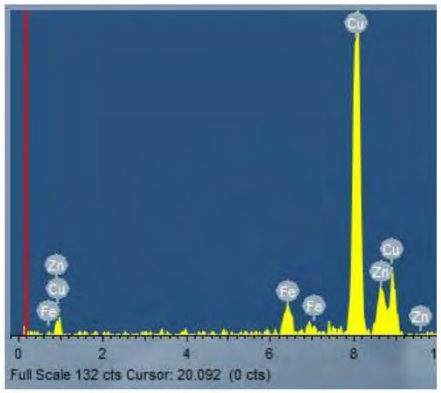
Crystalline lattice fringes (0.256 nm). ZnO (002) = 0.260 nm

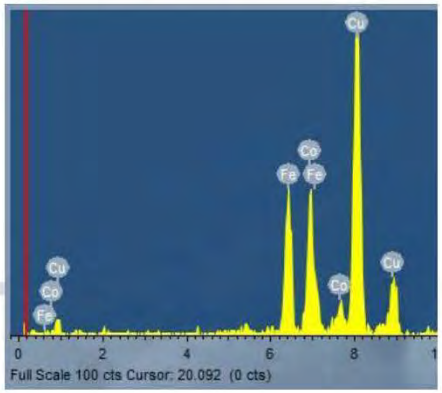
Particle is nominally a single crystal.

Witness Plate Post Test: D2 Location 5 TEM - EDS





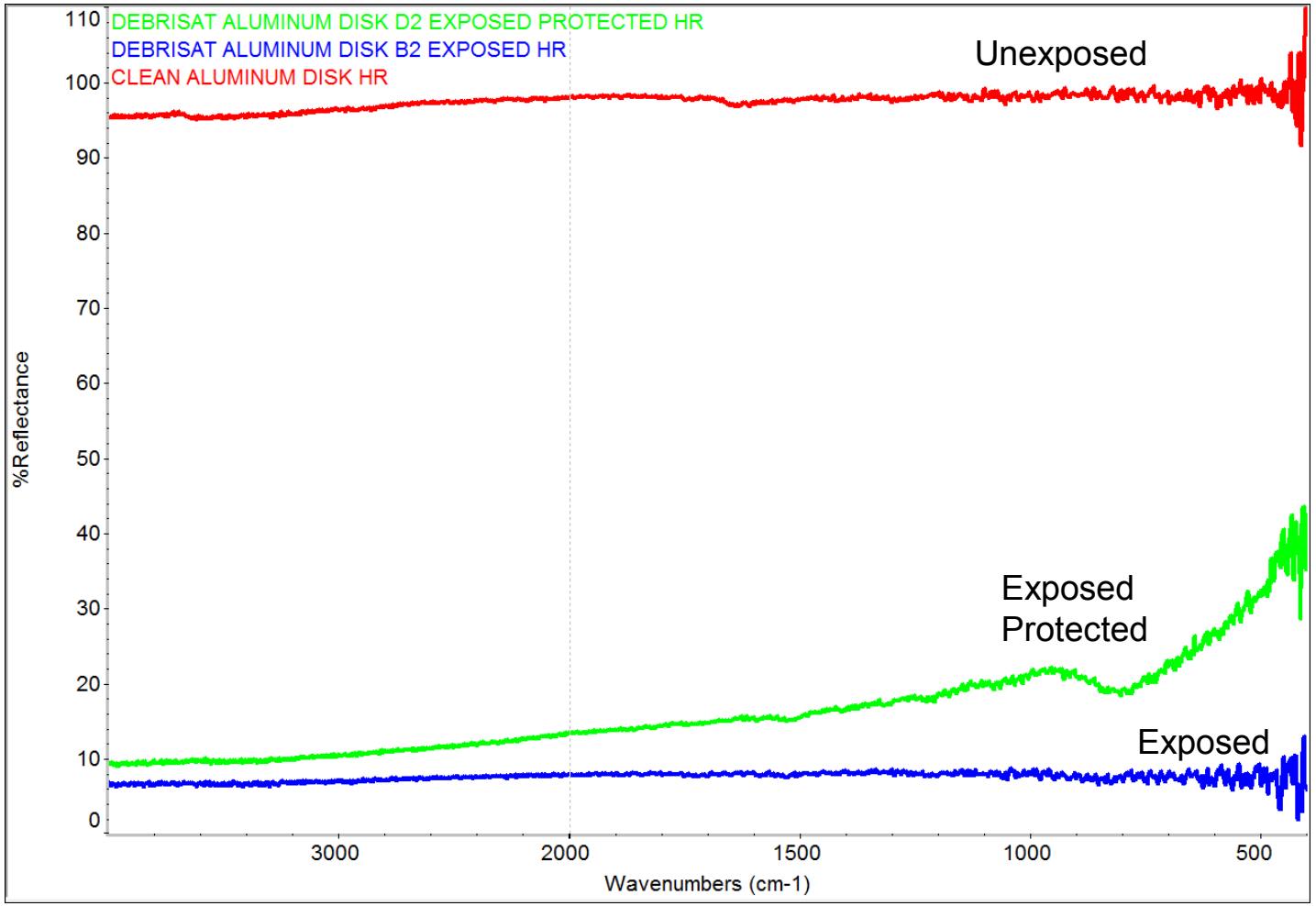




Particulates contain significant Cu, Fe and Co

Witness Plate Post Test: Aluminum Disks (B2, D2)

Quantitative LWIR FTIR Hemispherical Reflectance

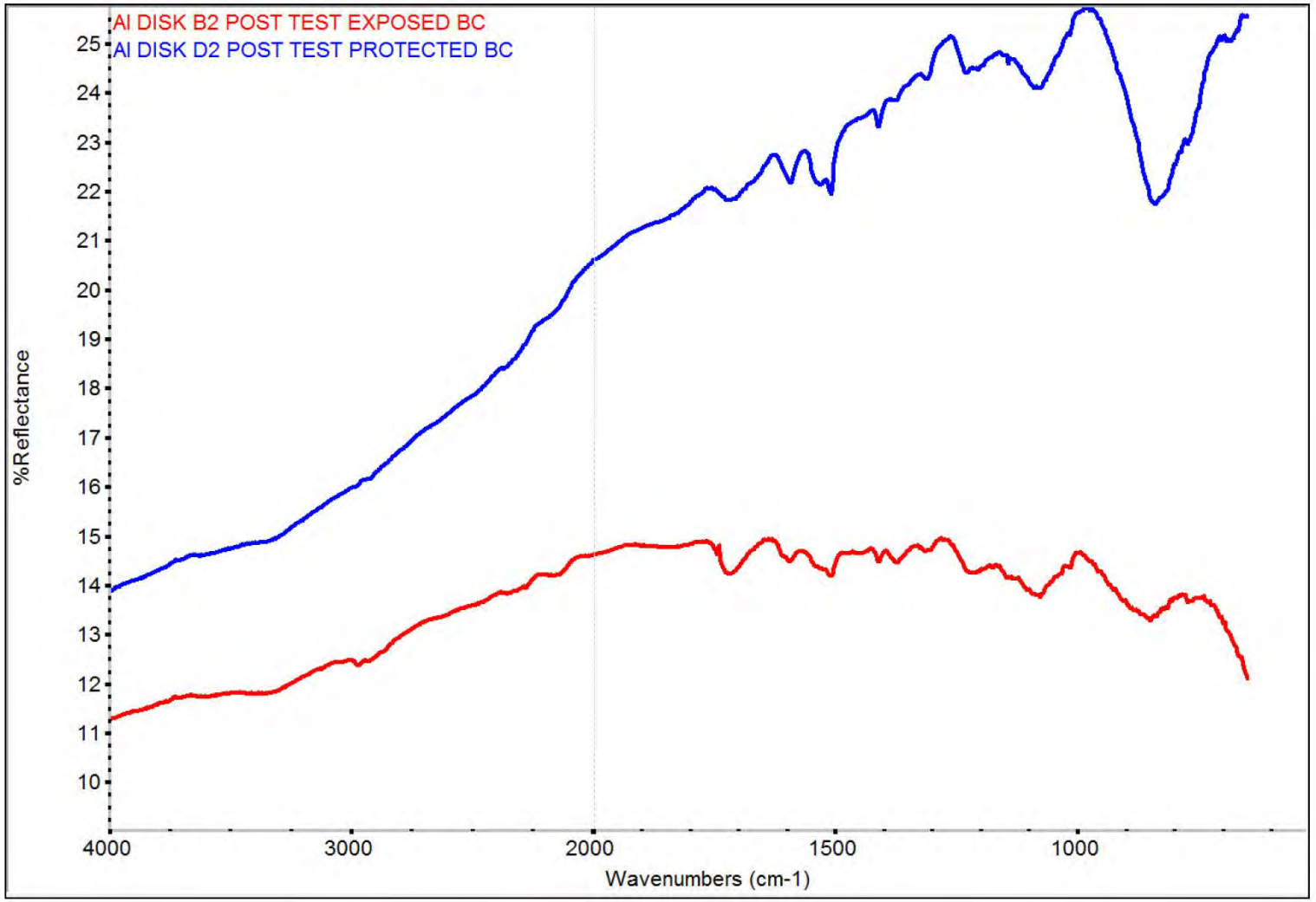


Significant darkening from 95% to 6% reflectance.

Witness Plate Post Test Aluminum Disks

SBIR Marking

Qualitative LWIR FTIR Biconical Reflectance

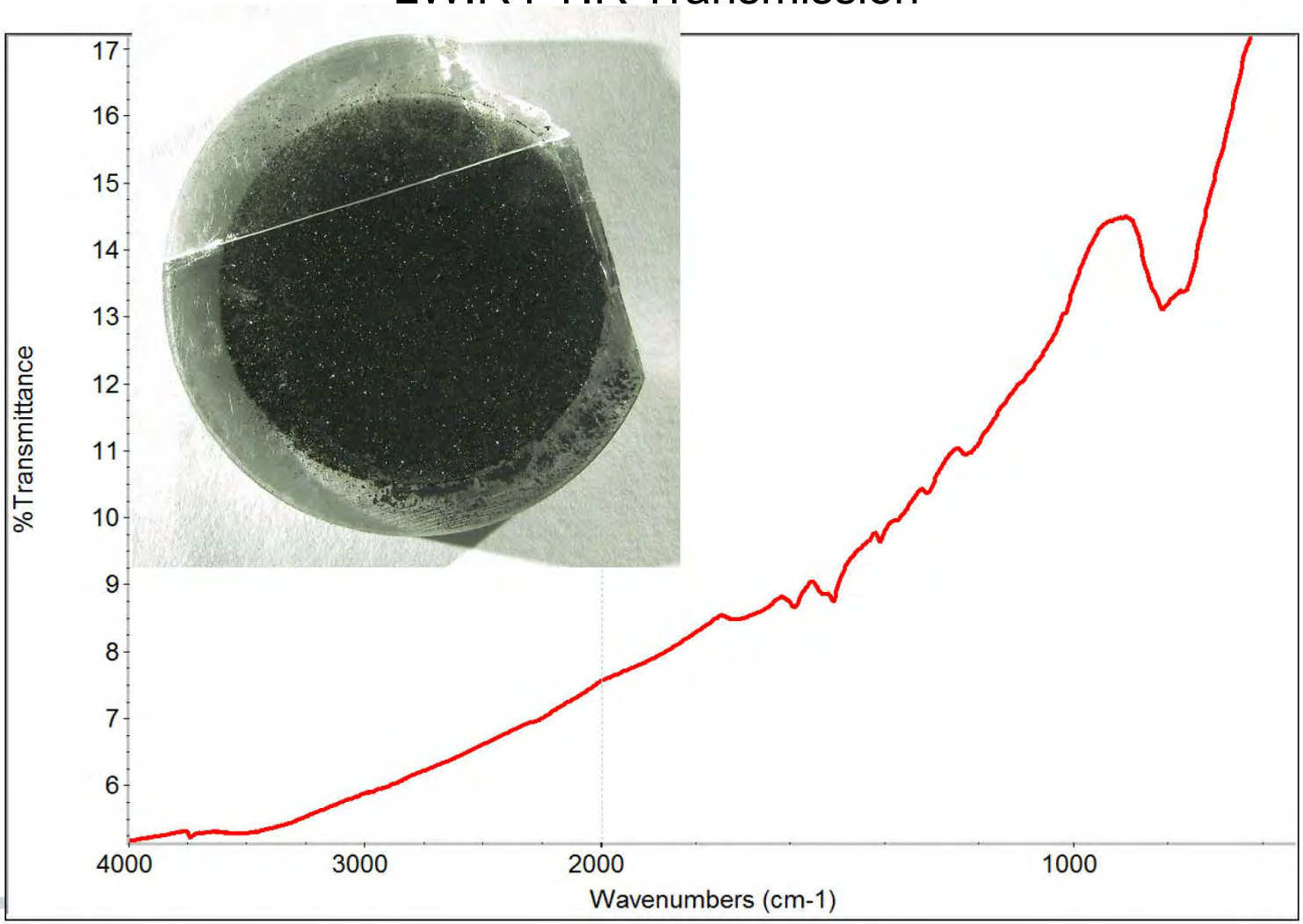


Soft catch contamination plus additional “oxide” band at 800 cm^{-1}. May be an aluminum oxide. Similar band seen in Debris-LV.

SBU Marking

Post Test: Protected NaCl Disk

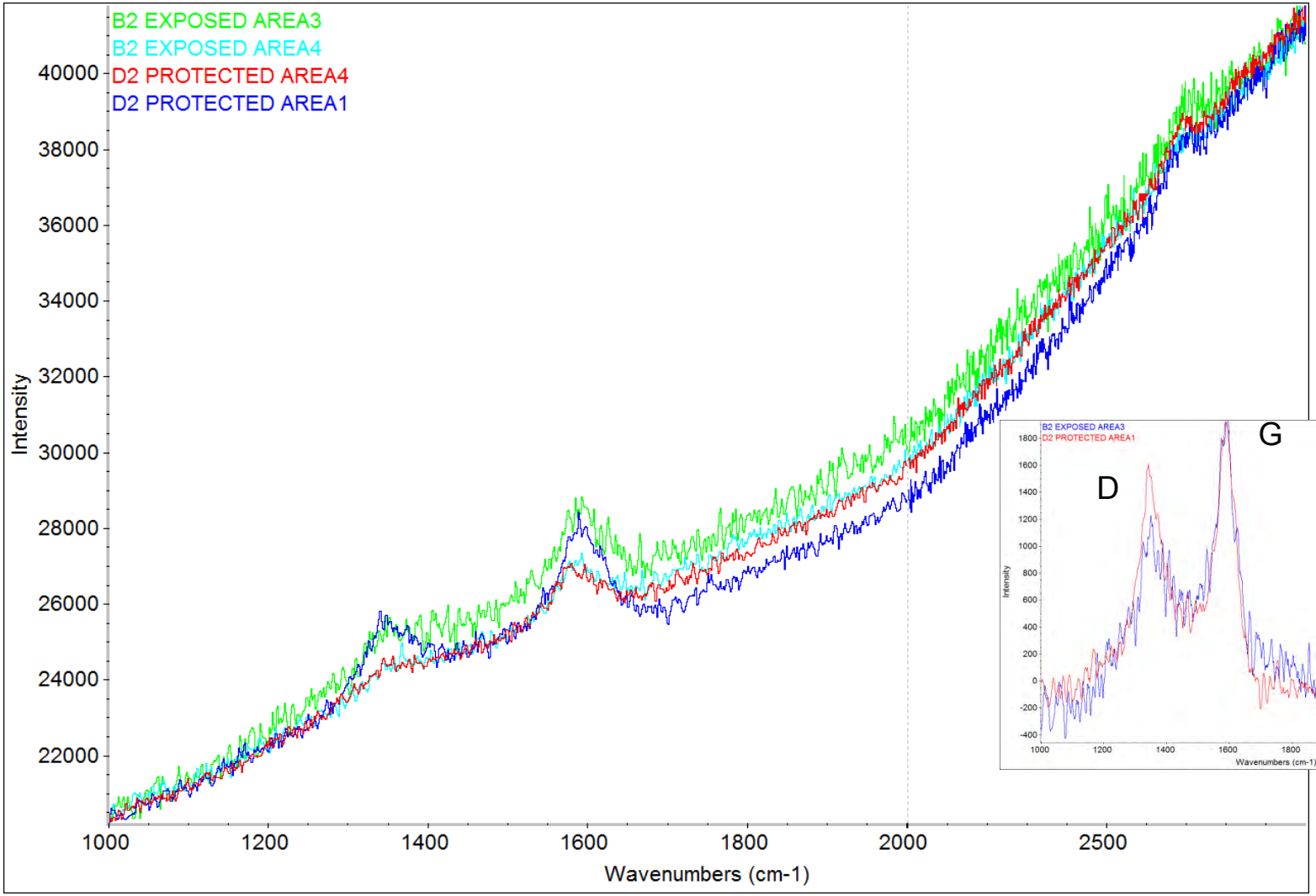
LWIR FTIR Transmission



Significant darkening with absorption band at 815 cm^{-1} similar to band seen by biconical reflectance.

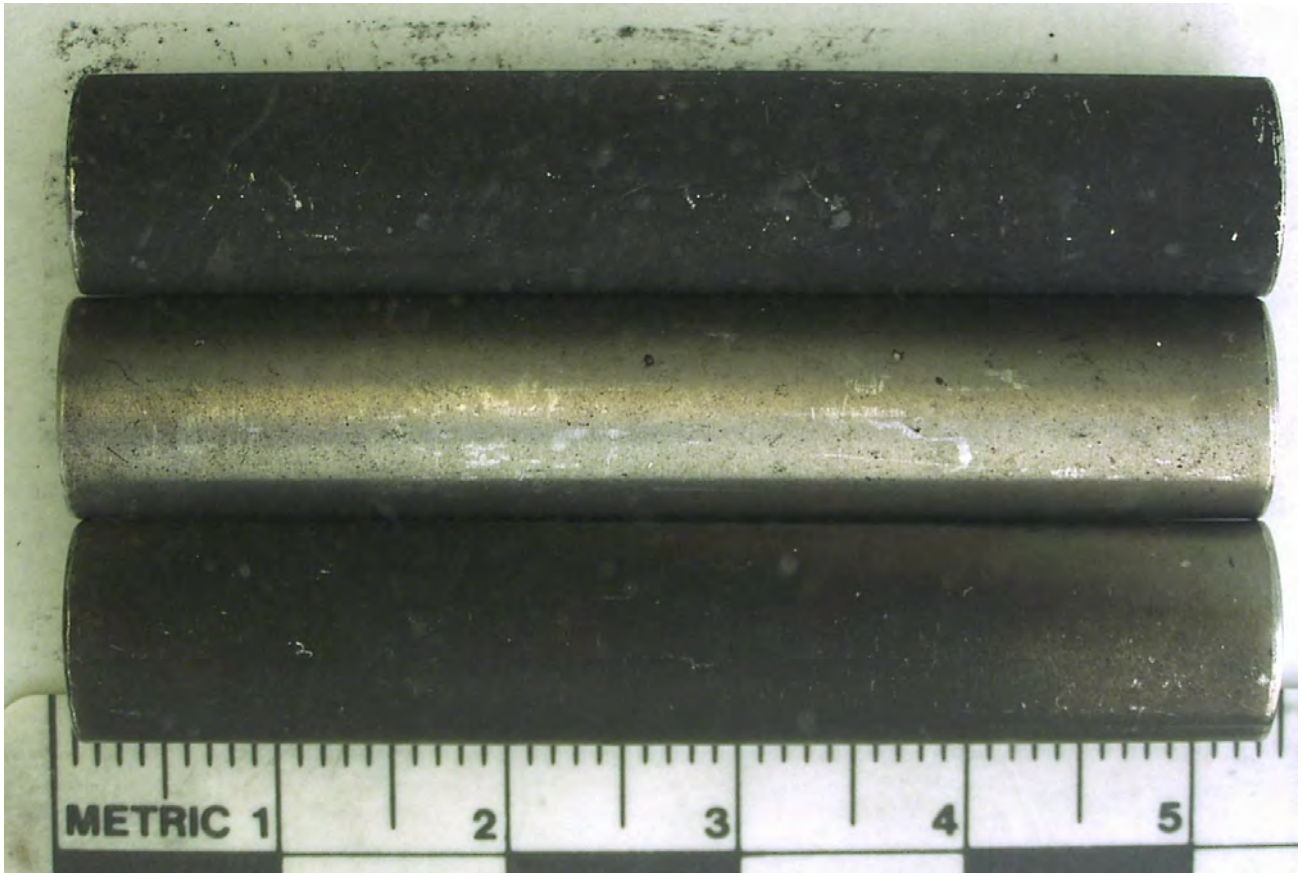
Witness Plate Post Test Al Disks

Raman Spectroscopy



Disordered (D) and crystalline graphite (G) bands observed in some areas. Similar to SEM stubs.

Witness Plate Whipple Plate Support Posts



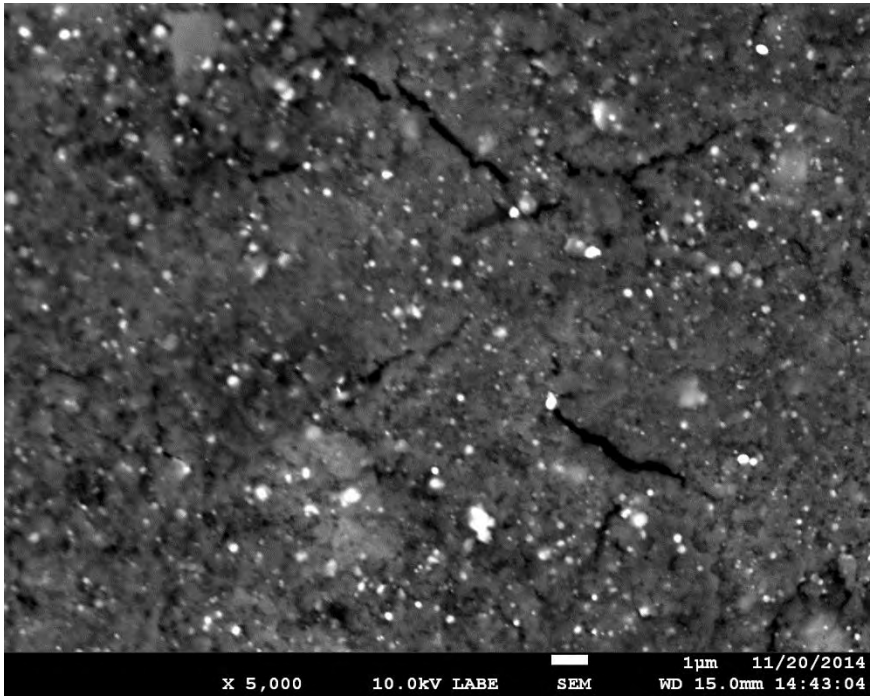
Unrinsed covered with black soot

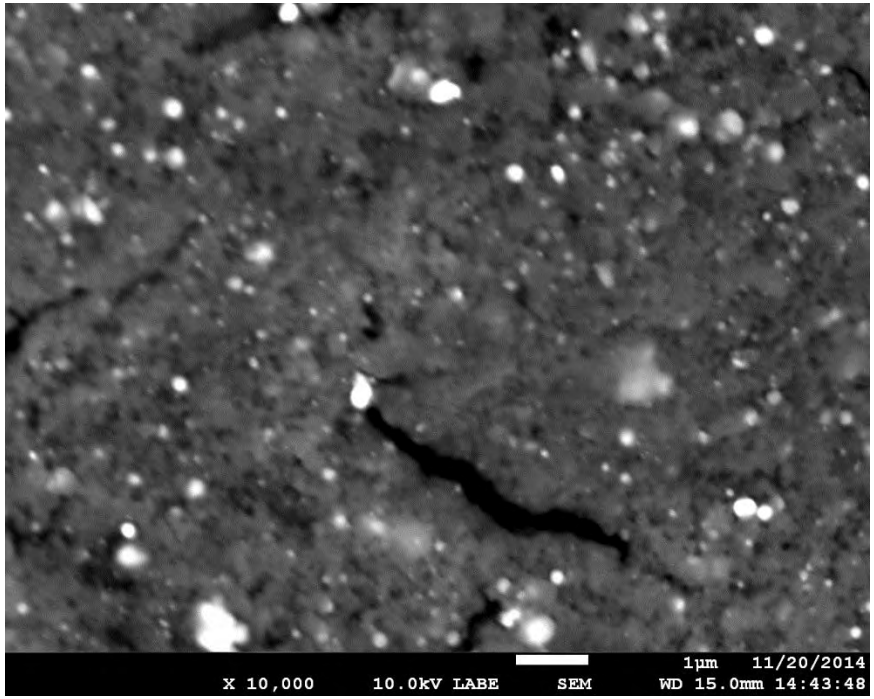
Rinsed Side 1

Rinsed Side 2

After rinsing with isopropyl alcohol the loose black soot is removed.
One side is relatively clean but a resistant black coating remains on one side indicating directional deposition.

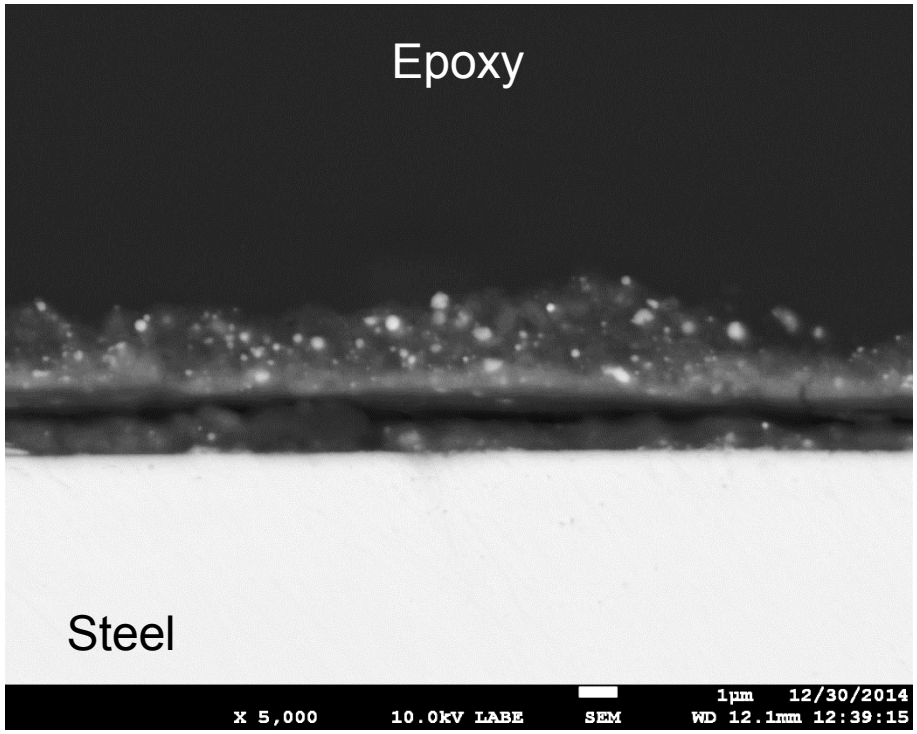
Rinsed Whipple Plate Support Post (surface)
Backscatter SEM 5KX, 10KX

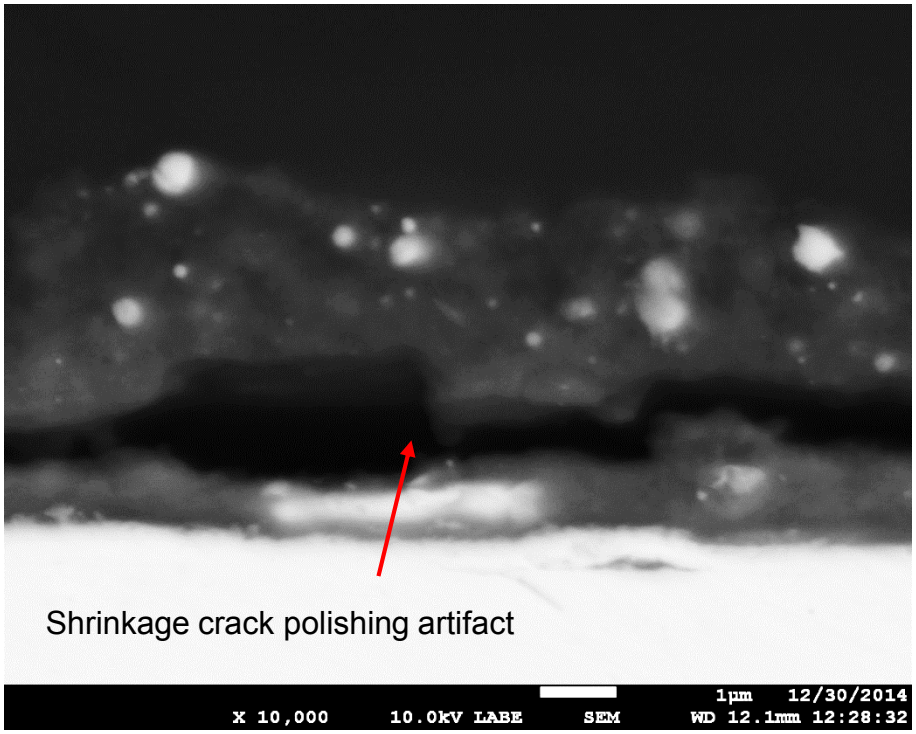




High Z (Al, Fe, Cu) nanoparticles embedded in carbonaceous matrix.

Rinsed Whipple Plate Support Post (polished cross section)
Backscatter SEM 5KX, 10KX

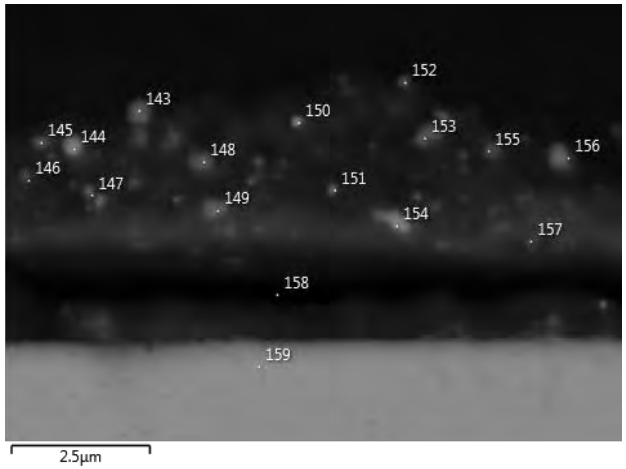


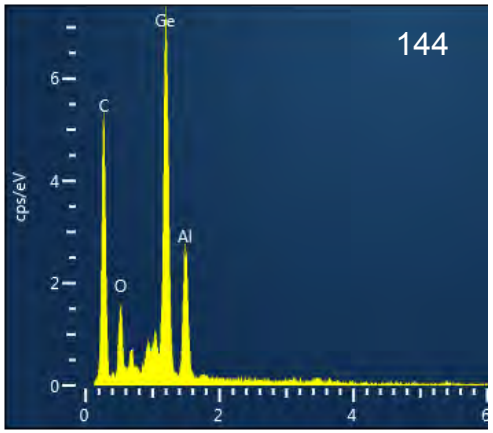


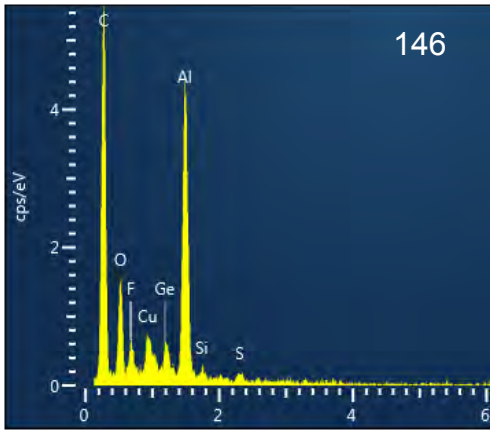
Metal nano droplets in a carbonaceous matrix 2-3 microns thick.

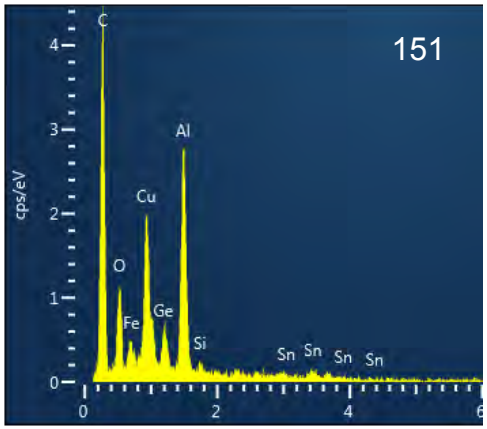
Rinsed Whipple Plate Support Post Deposit

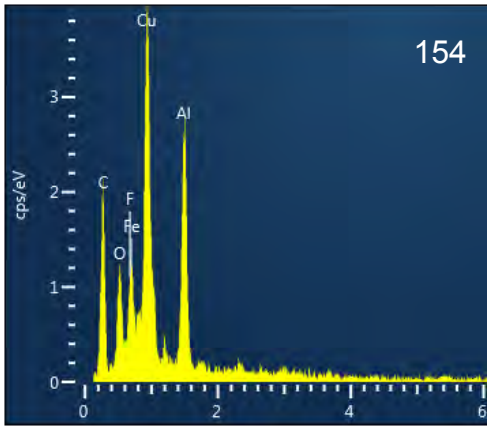
Backscatter SEM EDS Spectra

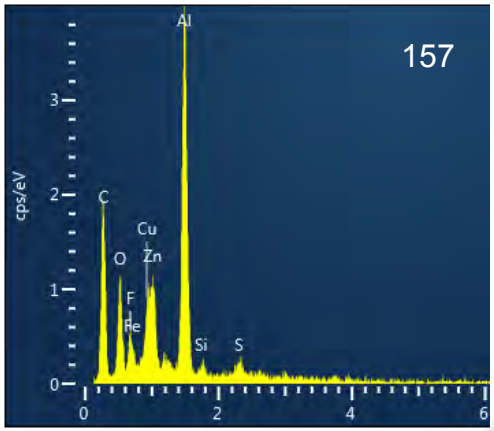










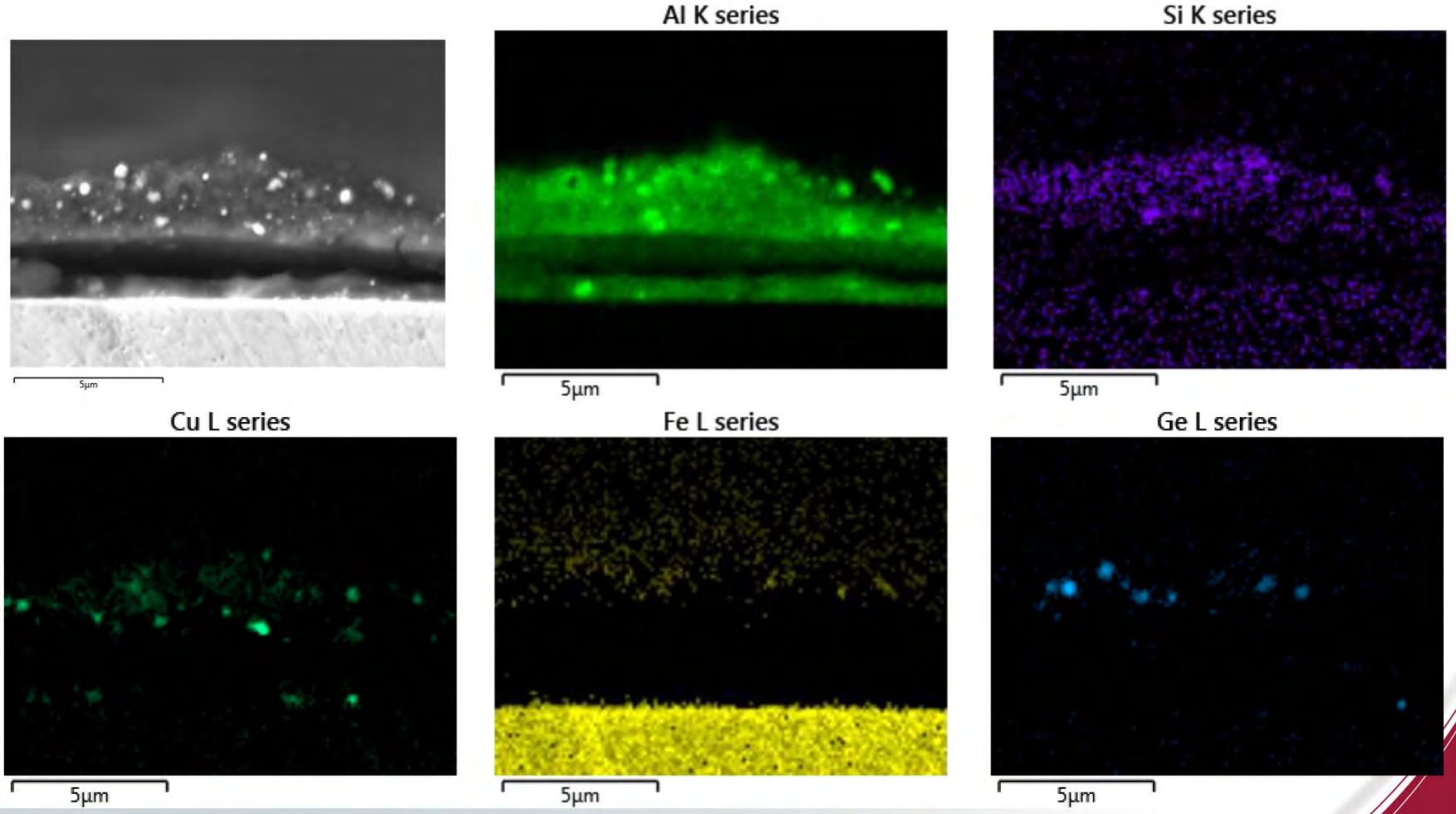


High Z nano particles in carbonaceous matrix contain Al, Cu, Ge, Fe and Zn.

SBU Marking

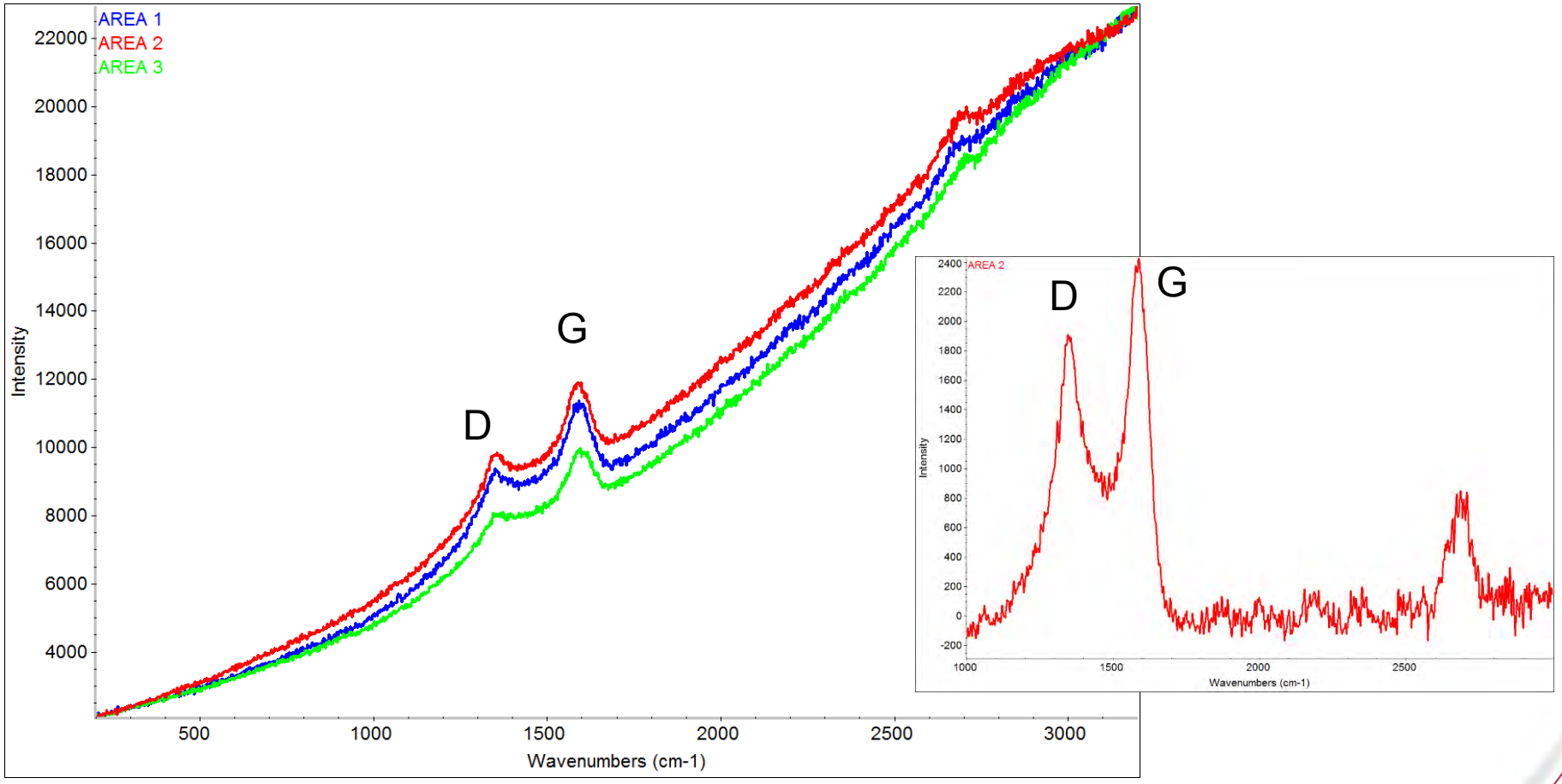
Rinsed Whipple Plate Support Post Deposit

Backscatter SEM and EDS maps



High Z nano particles in carbonaceous matrix contain Al, Cu, Ge, Fe,

Witness Plate Spacer: Raman Spectroscopy



Disordered (D) and crystalline graphite (G) bands observed on dark side of spacer.

Summary of Observations

- SEM stubs, witness plate assembly and DebriSat fragments were contaminated with soft catch fragments.
- SEM stubs also had a thin film of condensed soft catch vapor.
  - Similar to Debris-LV.
- Deposits on SEM stubs and witness plate assembly are predominantly C and O and consist of agglomerates of nano carbonaceous material.
  - Deposits are primarily from the soft catch – similar to Debris-LV
  - C-C composite honeycomb face sheets and MLI are also possible sources.
- Disordered graphitic carbon is present based on Raman spectra and TEM lattice fringe images. Similar to Debris-LV.
- The witness plate assembly was covered in a layer of loose “soot”, even under the protective Whipple plates.
  - Removal of the loose material by rinsing with isopropyl alcohol revealed a thin adherent coating on one side of the support posts indicating early directional deposition from DebriSat.
  - Coating was carbonaceous (disordered graphite) with nano metal particles.

Summary of Observations (cont.)

- Fluorine from Teflon wire insulation is also common.
- Nano droplets of metallic materials (Al, Fe, Cu, Zn, Ge) are also present indicating melting as a result of impact. Nano metal droplets also seen in Pre Preshot and Debris-LV.
  - Al is from the Al honeycomb, nadir and zenith panels, structural core and COPV liner.
    - Aluminum oxide particles are also present.
    - Al projectile is another source of aluminum.
  - Fe is from SS tubing and solenoids.
  - Ge is from the solar cells
  - Cu is from wiring and solenoids
- Solidified molten nano metal droplets are crystalline based on TEM lattice fringes and consist of only a few crystallites.

Summary of Observations (cont.)

- Witness plates show a significant decrease in reflectance (95% to 6%)
- Soft catch contamination seen in LWIR reflectance spectra of DebriSat fragments, SEM stubs and witness plate.
  - Not possible to get a clean spectrum of the debris generated by the hypervelocity impact
  - Additional “oxide” band at 800 cm^{-1} seen on some samples.
- Other laboratory analyses documented:
  - Aerospace TOR-2014-03201, Time-resolved Spectroscopy of Hypervelocity Impact Flash on DebriSat, Gouri Radhakrishnan.
  - Aerospace ATM-2014-03659, DebriSat Hypervelocity Impact Fragmentation Modeling, Naoki Hemmi

Appendix 1

Supplemental Information and Analyses

Introduction (cont.)

- Pre Preshot was conducted February 2014.
  - Validated performance of projectile to meet velocity goal of 7 km/s.
  - Confirmed operational status of test chamber and facility.
  - Target was primarily designed to catch the projectile.
    - Multi-shock shield supplied by NASA.
    - Multiple bumper panels of fiberglass, stainless steel mesh and Kevlar.
  - **No “soft catch “ panels were installed** (unlike Debris-LV and DebriSat).
  - Test conducted with a pressure of ~1-2 Torr nitrogen.
  - A witness plate assembly was provided by Aerospace in order to catch and sample debris.
- Additional Documentation
  - Aerospace TOR- 2014-03082, DebriSat Pre Preshot Laboratory Analyses, P. M. Adams and P. M. Sheaffer, December 23, 2014.

Introduction (cont.)

- Debris-LV (Pre Shot) conducted 1 April 2014
  - Further validated performance of projectile and facility and served as a dress rehearsal for the DebriSat test.
  - The 15 kg target consisted primarily of empty tanks and was constructed by Patti Sheaffer from materials representative of a launch vehicle (LV) upper stage.
    - Primarily aluminum and titanium with lesser amounts of copper and stainless steel.
  - Test chamber was lined with “soft catch” foam panels to trap fragments for size distribution analysis.
  - A witness plate assembly was constructed by Aerospace in order to catch and sample debris and returned to Aerospace after the test for analysis.
  - Aerospace also placed SEM stub witness plates into soft catch for post test retrieval and analysis.
- Additional documentation
  - Aerospace TOR-2014-03577, Debris-LV Hypervelocity Impact Post-Shot Physical Results Summary, P. M. Sheaffer.
  - Aerospace TOR-2015-00928, Debris-LV Laboratory Analyses, P. M. Adams, P. M. Sheaffer, Z. R. Lingley and G. Radhakrishnan.

Laboratory Instrumentation

- Field Emission Scanning Electron Microscopy (FESEM)
  - JEOL JSM-7600F SEM.
  - In-lens secondary electron detector (SEI mode).
    - High resolution imaging.
  - Lower secondary electron detector (LEI mode).
    - Less charging.
    - Enhances topography.
  - Backscatter electron detector (LABE mode)
    - Atomic number (Z) image contrast.
- Energy Dispersive (X-ray) Spectroscopy (EDS) in the SEM/TEM
  - Oxford X-Max silicon drift detector (SEM).
  - Oxford SiLi detector (TEM)
- Transmission Electron Microscopy (TEM)
  - JEOL JEM-3100F TEM.
  - Oxford INCA EDS.

Laboratory Instrumentation (cont.)

- Fourier Transform Infrared (FTIR) spectroscopy
  - Nicolet 6700 spectrometer.
  - Harrick Scientific “praying mantis” diffuse reflectance accessory.
    - Qualitative reflectance.
    - Mercury cadmium telluride (MCT) detector.
    - Fast analysis with excellent signal to noise.
    - Can only analyze small samples (< 1”).
  - Labsphere hemispherical reflectance accessory.
    - Quantitative reflectance.
    - Long scan time with poor signal to noise.
- Raman Spectroscopy
  - Renishaw inVia Raman microscope.
- UV-VIS-NIR Spectroscopy
  - Perkin Elmer Lambda 900 Spectrometer
    - Diffuse transmission and reflectance with integrating sphere.

Considerations for SEM and EDS Analyses

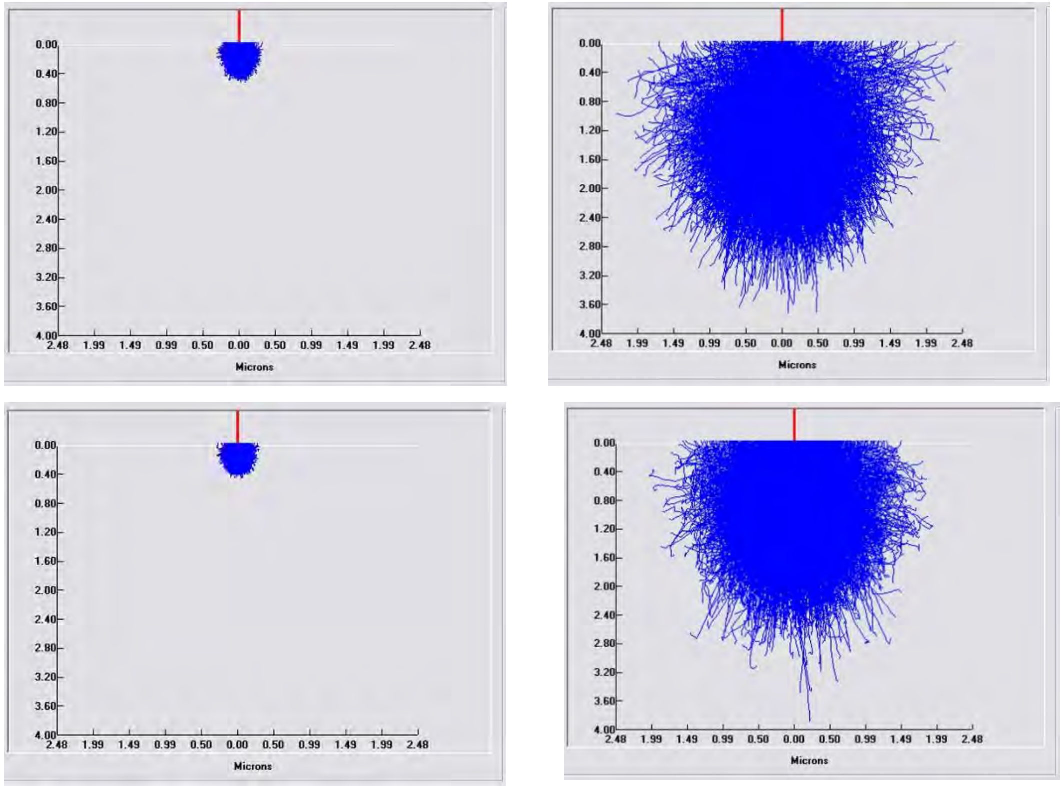
- A preliminary look at Debris-LV and DebriSat SEM stubs indicated that a thin coating, containing low atomic number (Z) materials (carbon), was present.
- This presented challenges and options for SEM-EDS analyses. Low vs. high voltage (5 KV vs. 15 KV).
- Advantages of 5 KV: high resolution imaging of thin low Z surfaces, EDS from surface material and not substrate.
- Disadvantages of 5 KV: beam does not penetrate beneath the surface (subsurface is hidden), does not excite X-rays from medium Z elements (Cl to Ti), high Z element K series not excited but L series X-rays are.
  - Most medium Z elements not expected .
- Advantages of 15KV: Can see a limited distance beneath the surface, excites X-rays from just about all elements.
- Disadvantages of 15 KV: loss of detail in low Z surfaces, for thin coatings may penetrate through to the substrate

<u>Use both 5KV to15 KV to obtain more comprehensive information.</u>

Electron Penetration into Materials

C

Al



5 KV

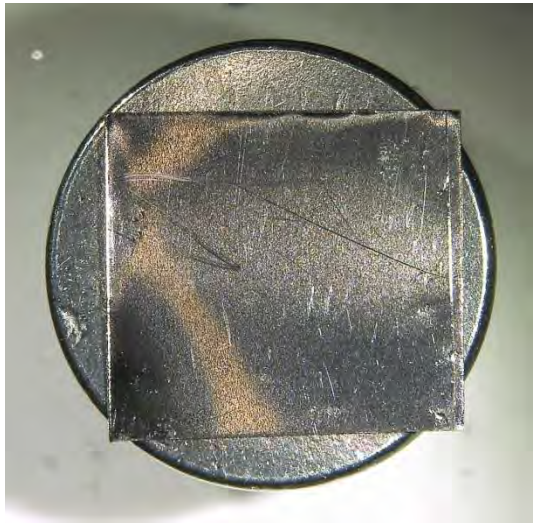
15 KV

•Electron penetration influences depth of X-ray generation (EDS).

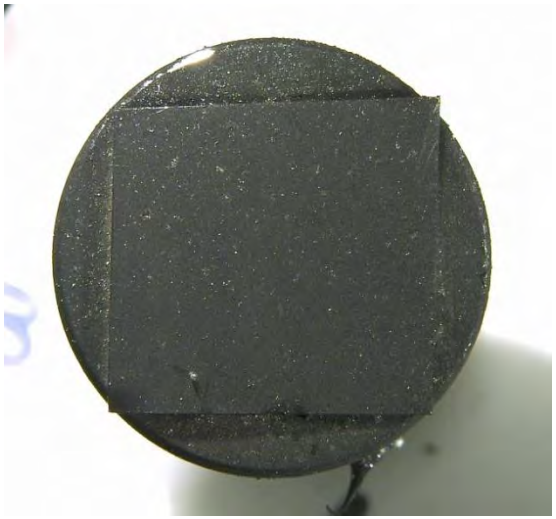
•Secondary emitted electrons have low energy and image primarily the surface.

•Backscattered emitted electrons have greater energy and image partially into the surface and have atomic number information.

SEM Stubs



Unexposed



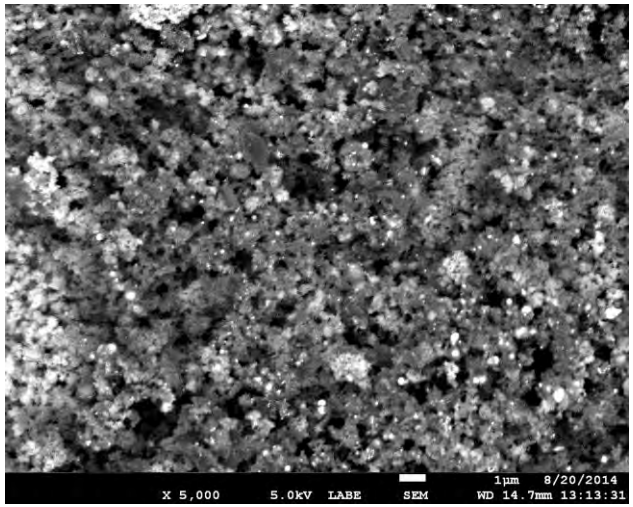
Exposed #13

Exposed #14

Note significant darkening of post test stubs

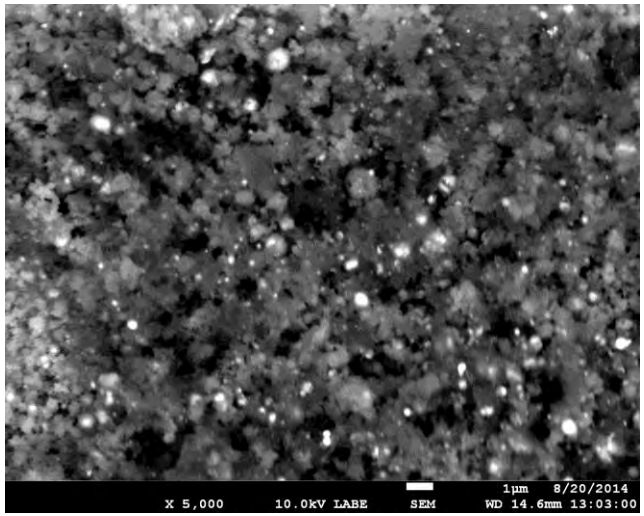
SEM Stub 4: Backscatter Electron SEM (5 KX)

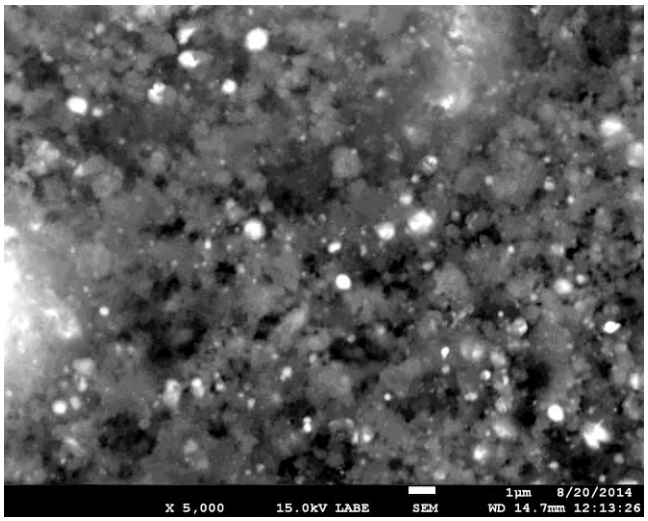
Brighter areas are higher atomic number (Z).



5 KV

10 KV

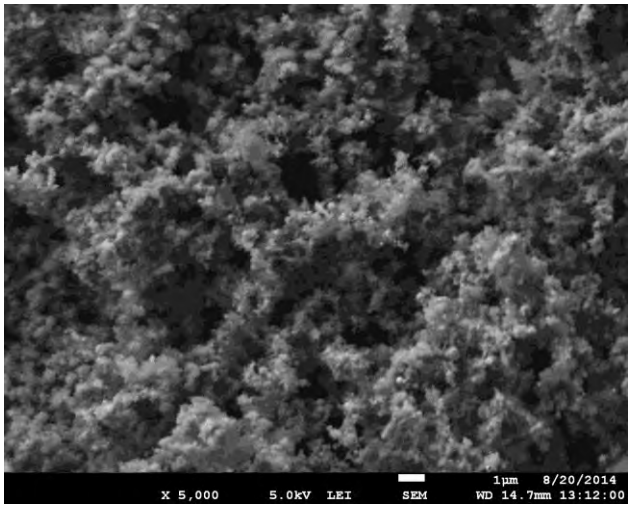




15 KV

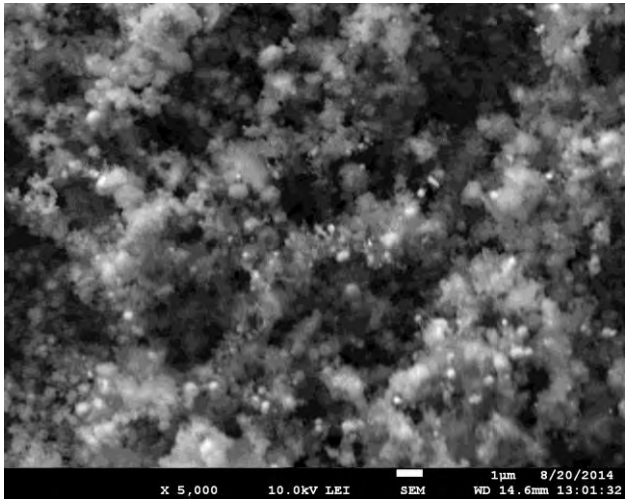
Images are from the same area.
Greater penetration and less surface detail with increasing energy.

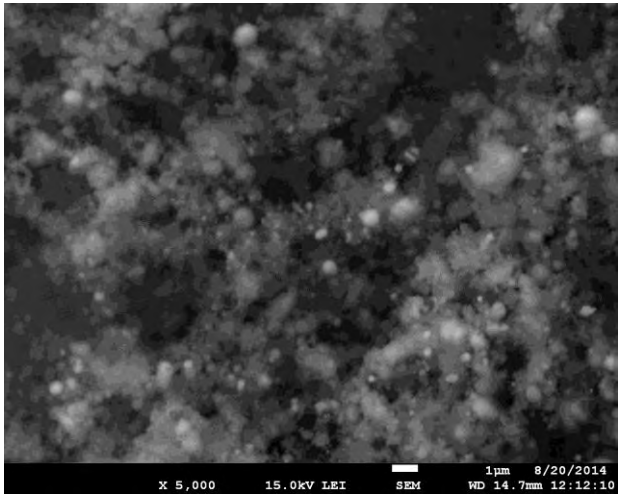
SEM Stub 4: Secondary Electron SEM (5KX)



5 KV

10 KV



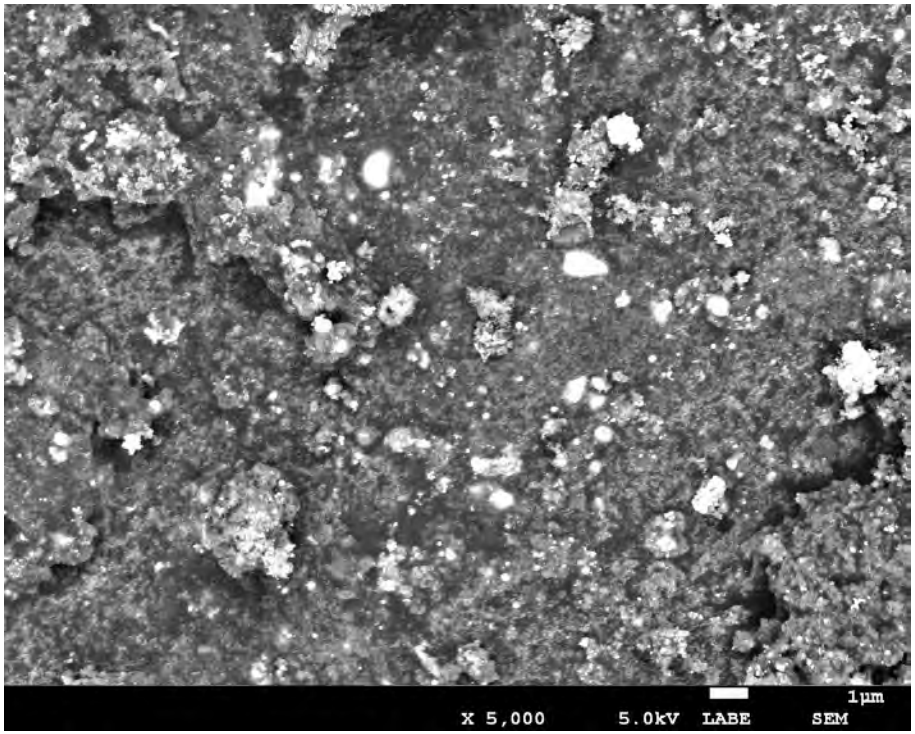


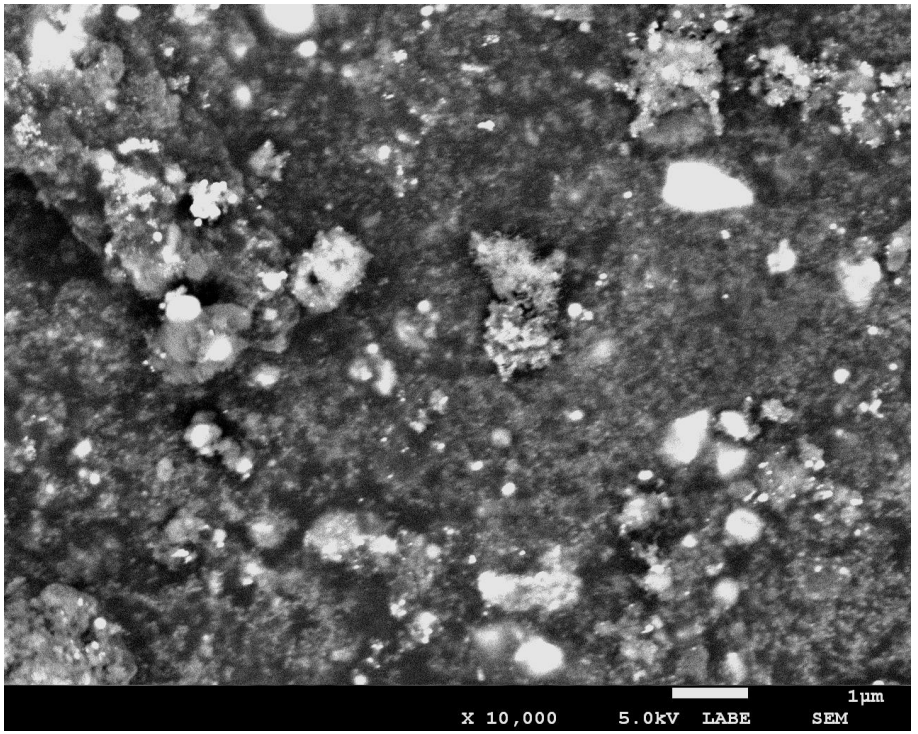
15 KV

Images are from the same area as previous slide.
Greater penetration and less of surface detail with increasing energy.

SEM Stub 13

Backscatter SEM (5KX, 10KX)



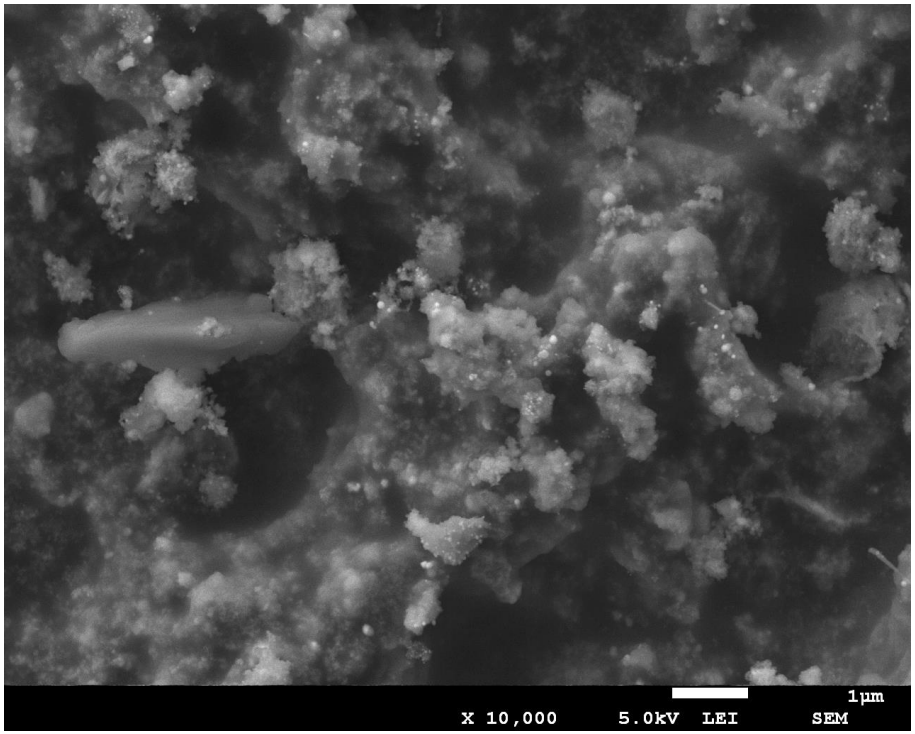


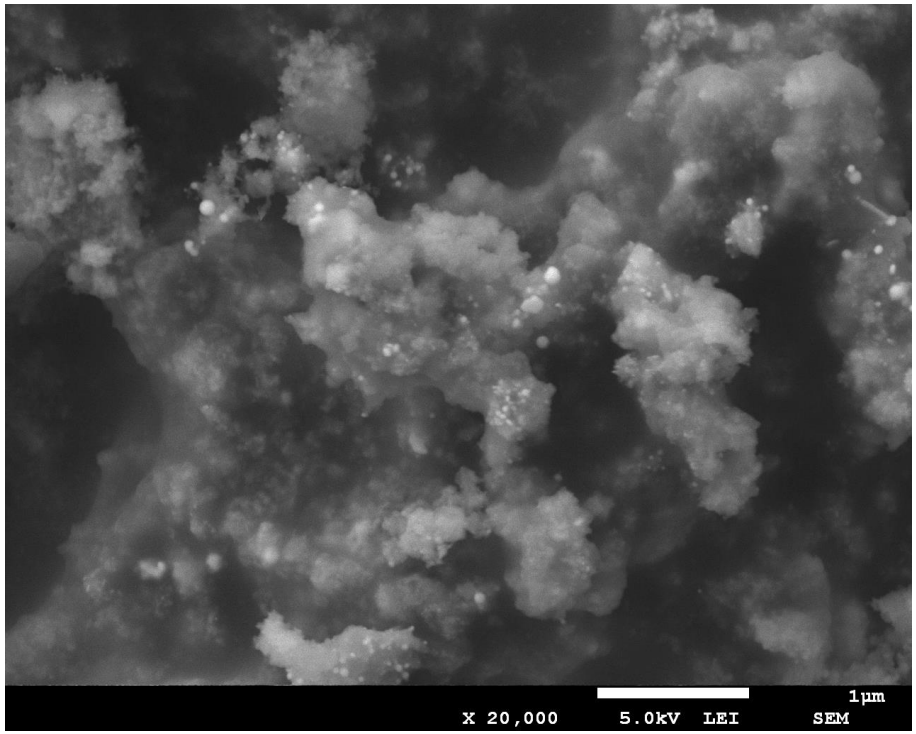
Material on surface consists of nano-sized agglomerates

Located 11 feet down range – 3 O'clock

SEM Stub 13

Secondary Electron SEM (10KX, 20KX)

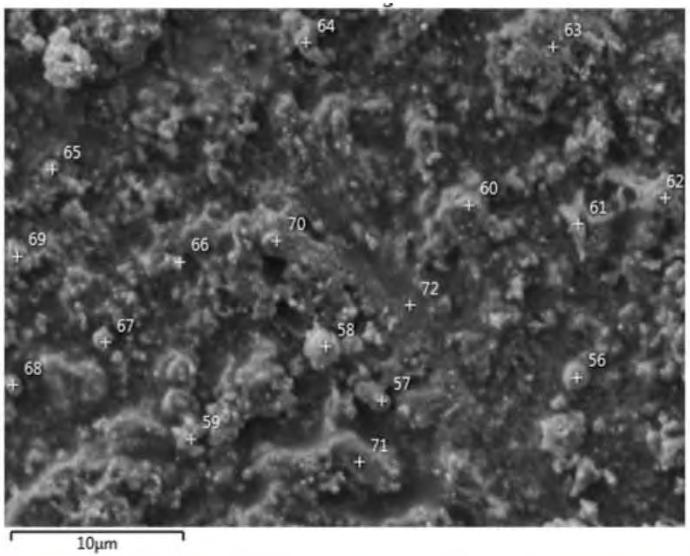


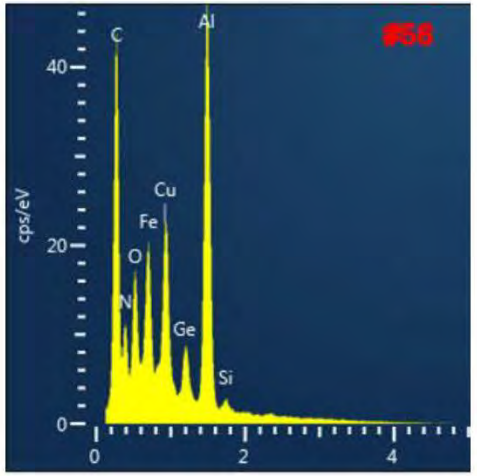


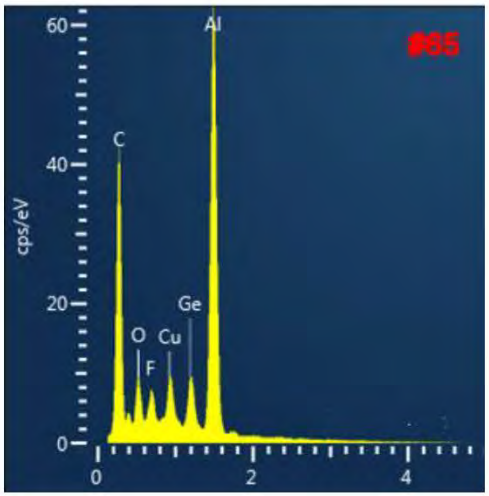
Note nano-scale structures

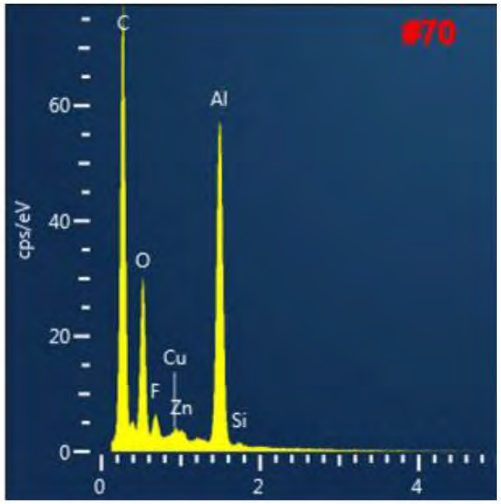
SEM Stub 13

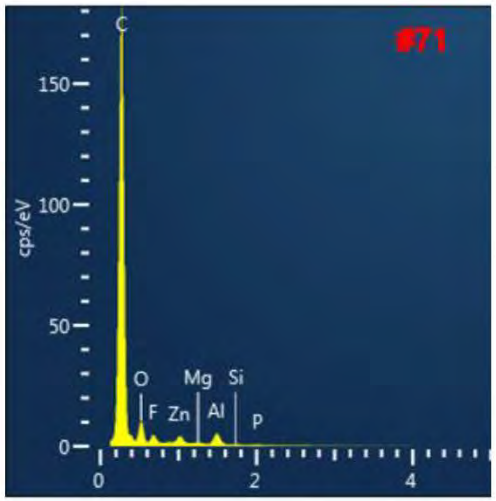
SEM EDS







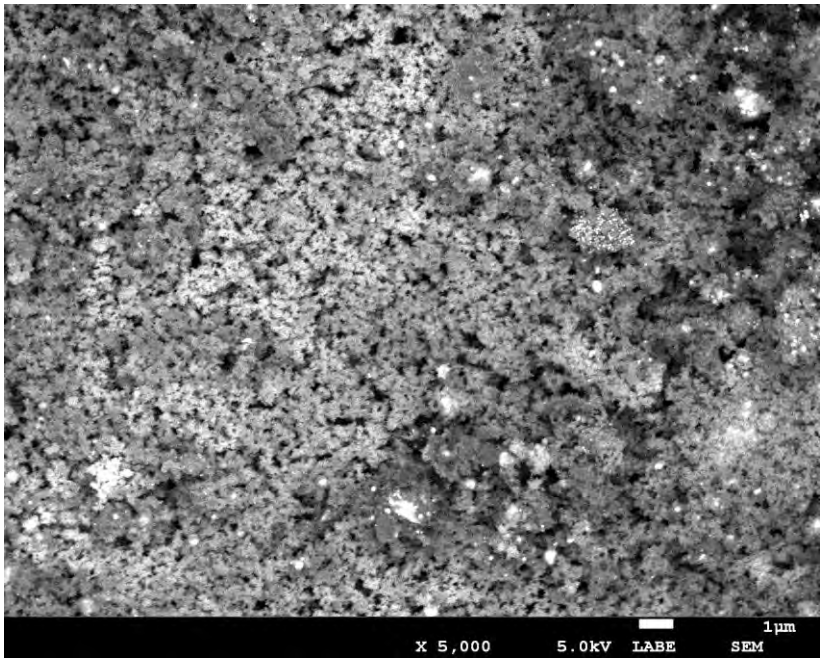


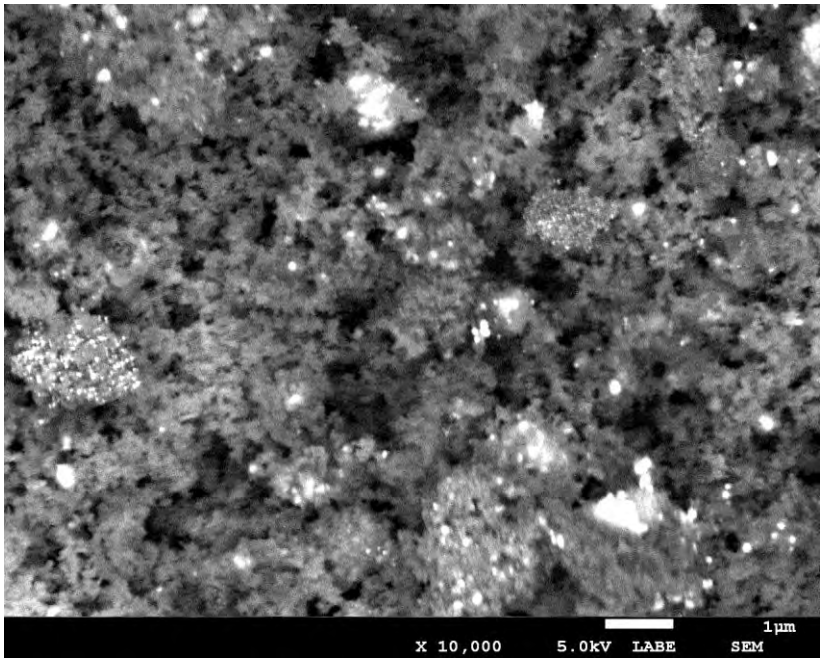


Al, C, Cu, O, Fe, and Ge are common.

SEM Stub 14

Backscatter SEM 5KX, 10KX



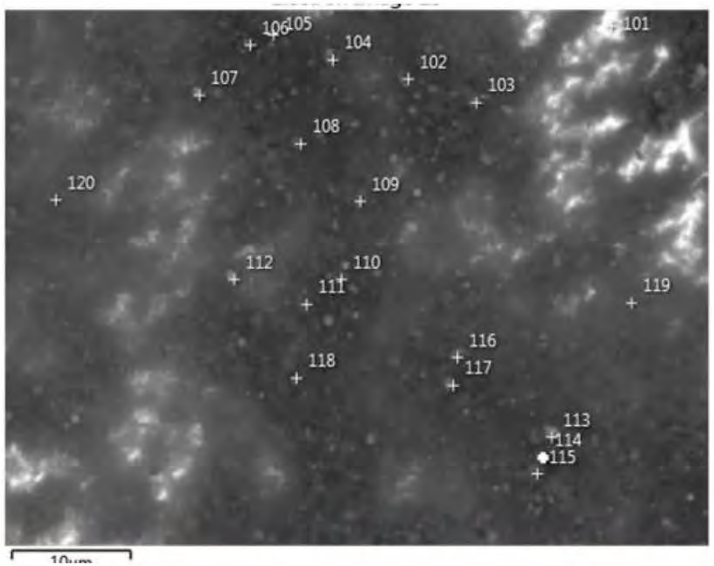


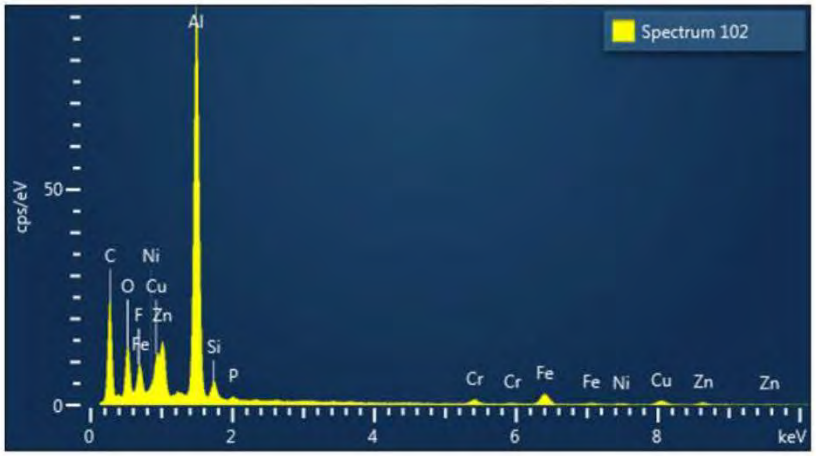
Material on surface consists of nano-sized agglomerates

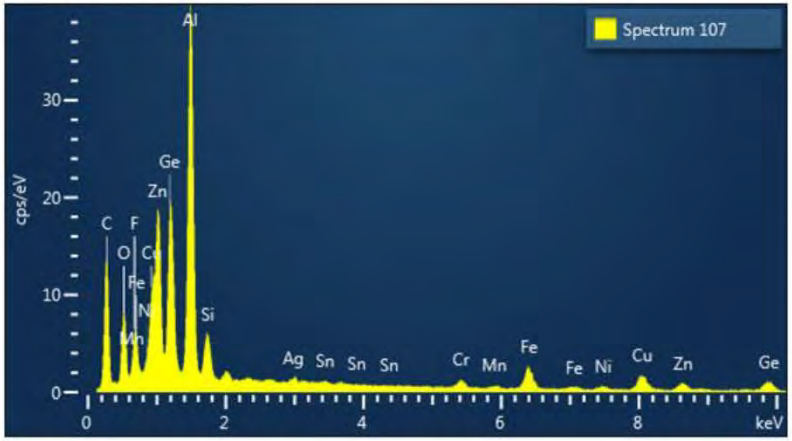
Located 15 feet down range – 3 O'clock

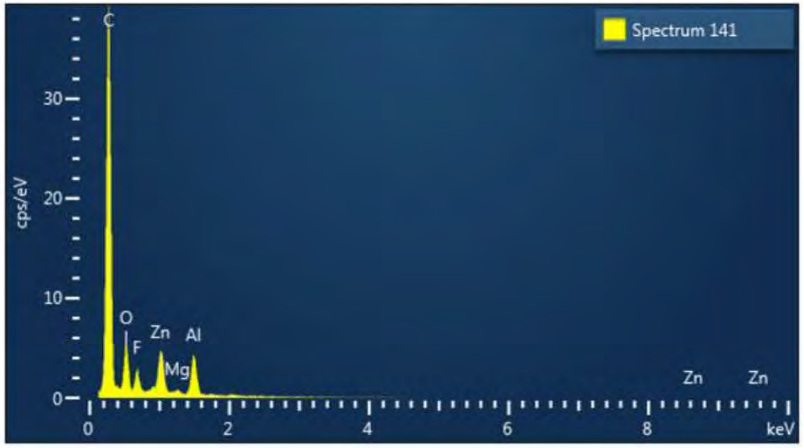
SEM Stub 14

SEM EDS (15 KV)







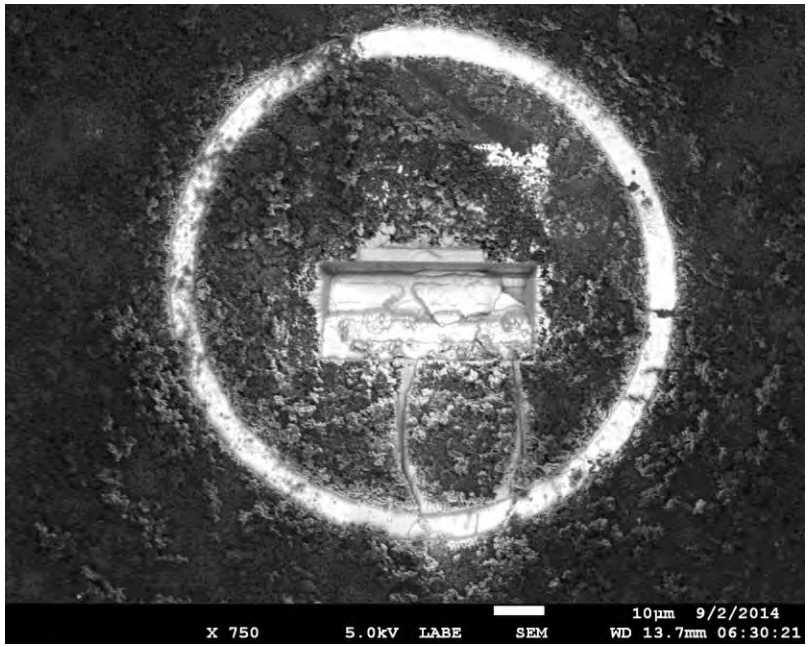


Al, C, Cu, O, Fe, Zn and Ge are common.

SEM Stub 14: Focused Ion Beam (FIB) Cross Section

Backscatter SEM

Circle trenched down to Ta to facilitate site location



Protective Pt layer deposition

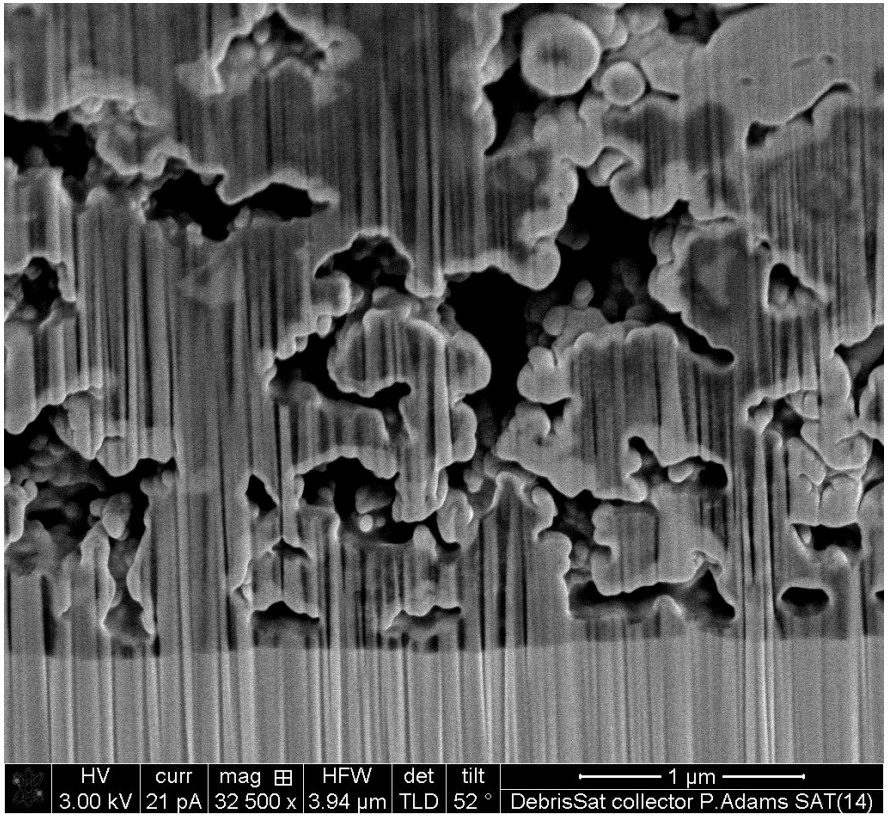
Vertical wall

50 deg angled trench

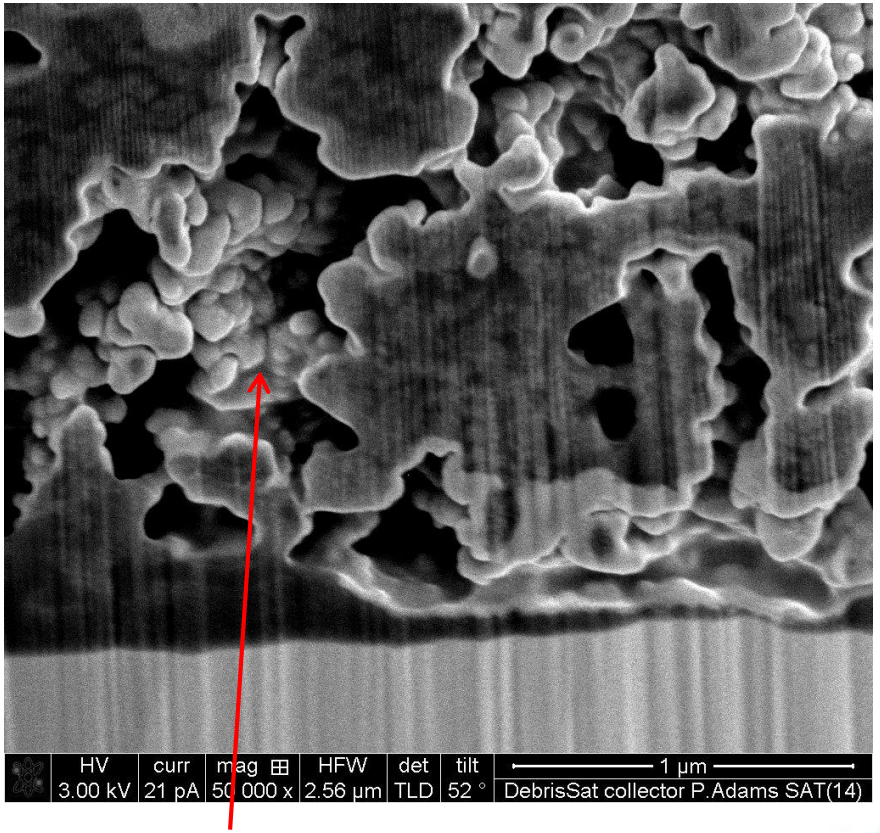
Plan View

SEM Stub 14: FIB Cross Section

SEM 32.5KX, 50 KX



Striations (curtains) are artifacts of FIB preparation.



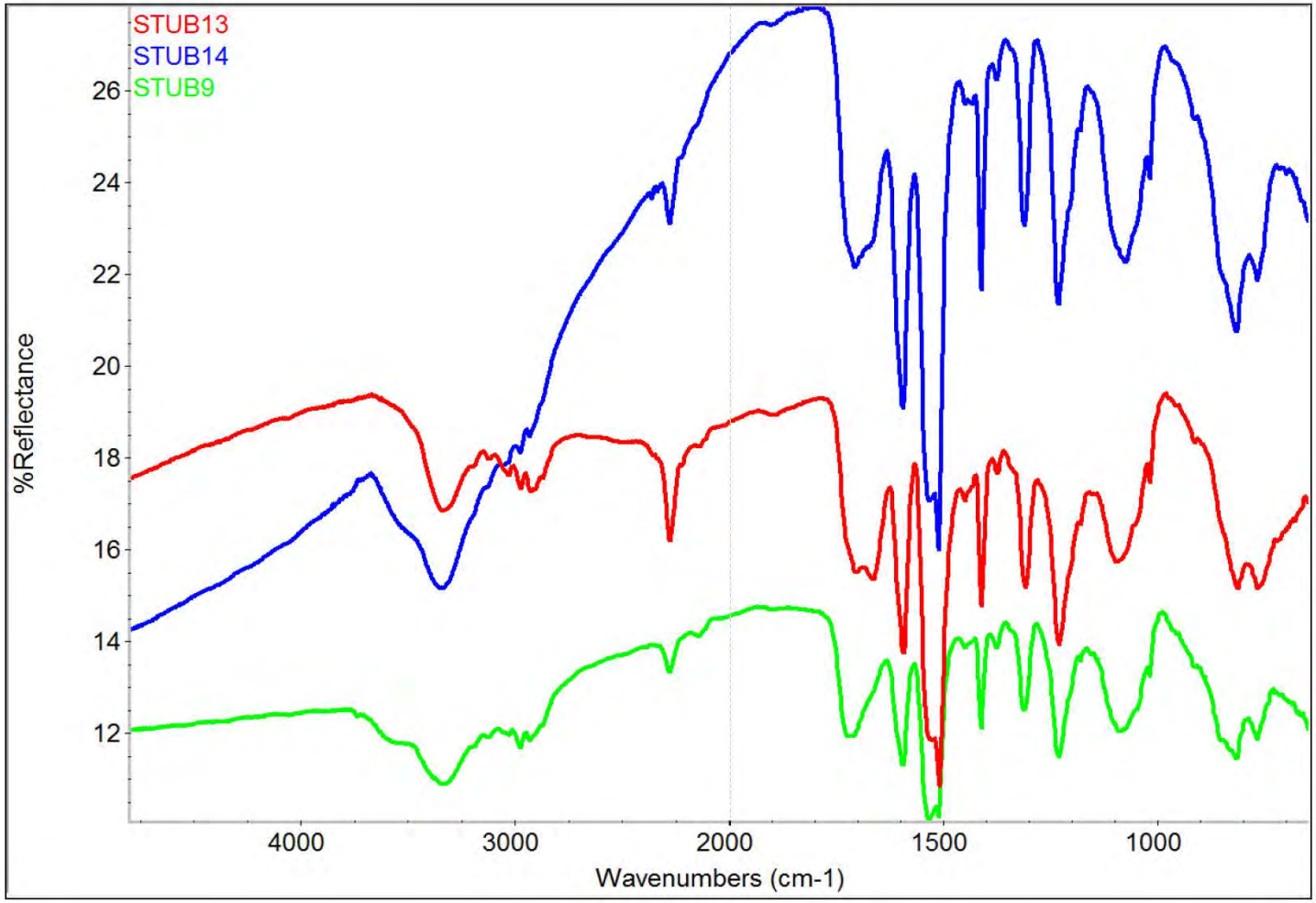
Back deposition of Ta from trenching into voids?

View of Side Wall

SBU Marking

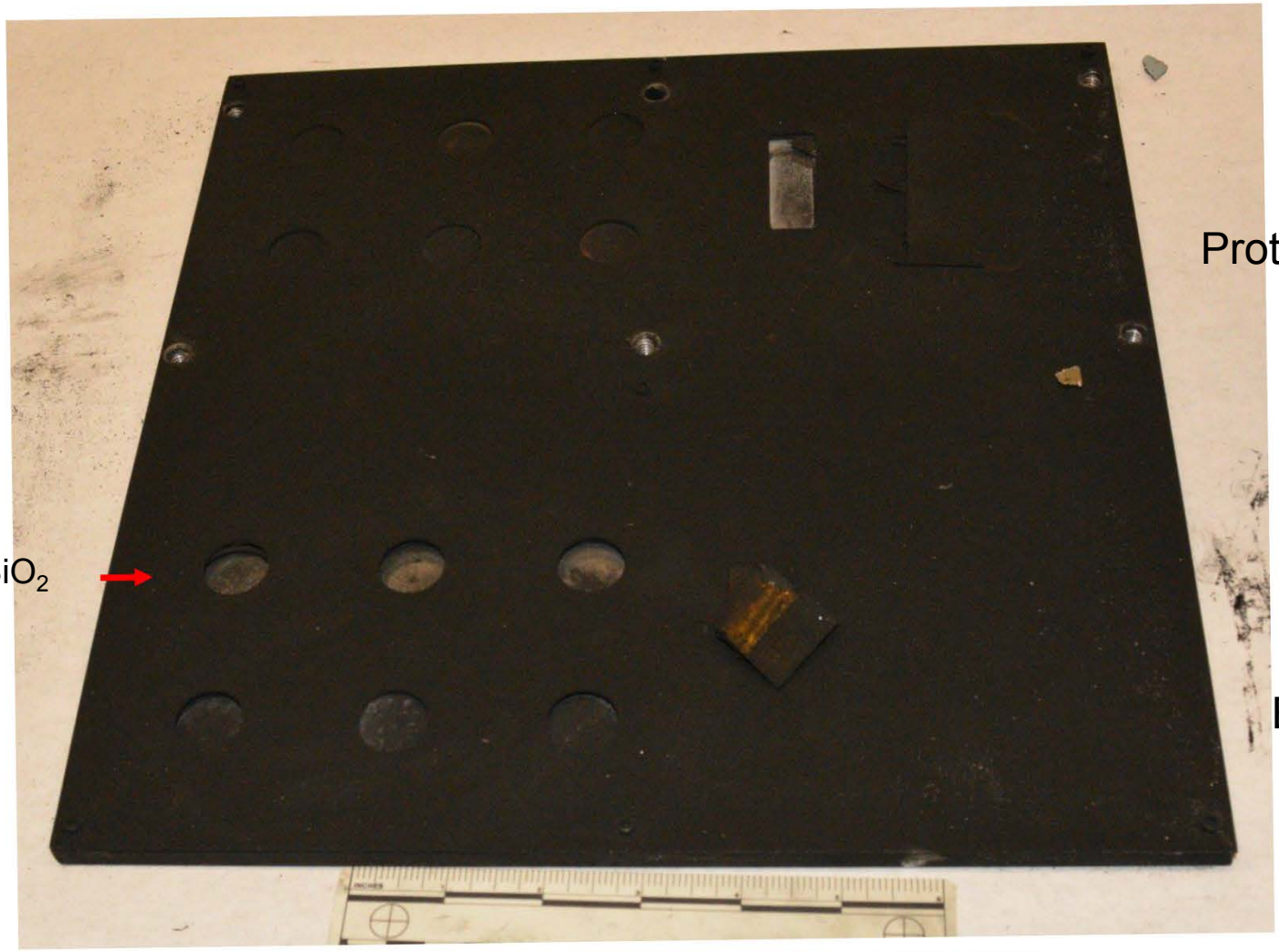
Post Test: SEM Stubs

FTIR – Qualitative Diffuse Reflectance



Significant soft catch signature is present on SEM stubs.

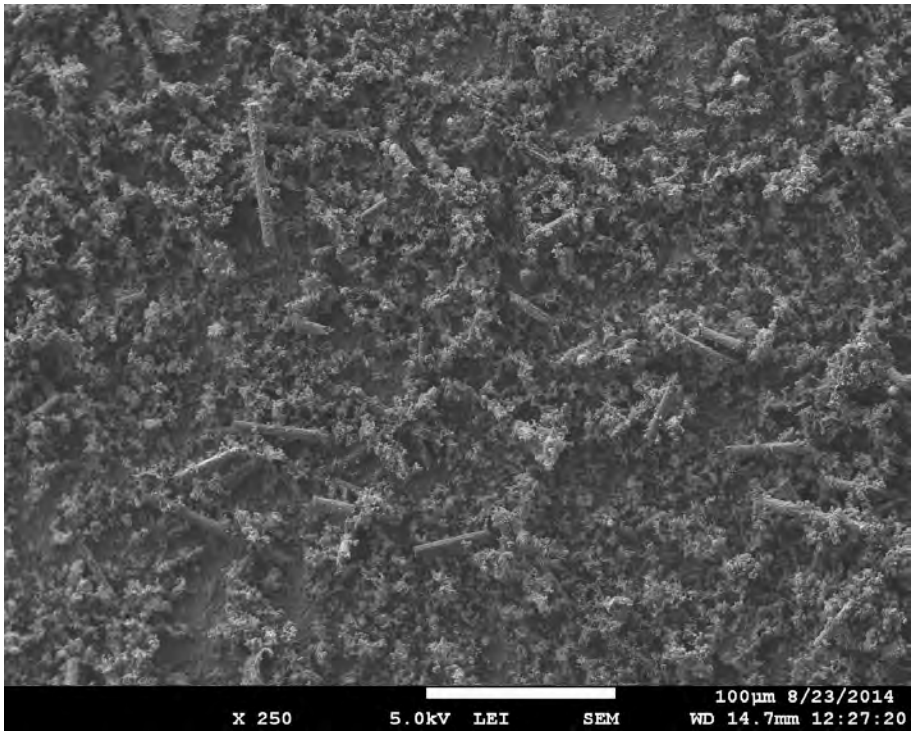
Witness Plate Assembly: Post Test

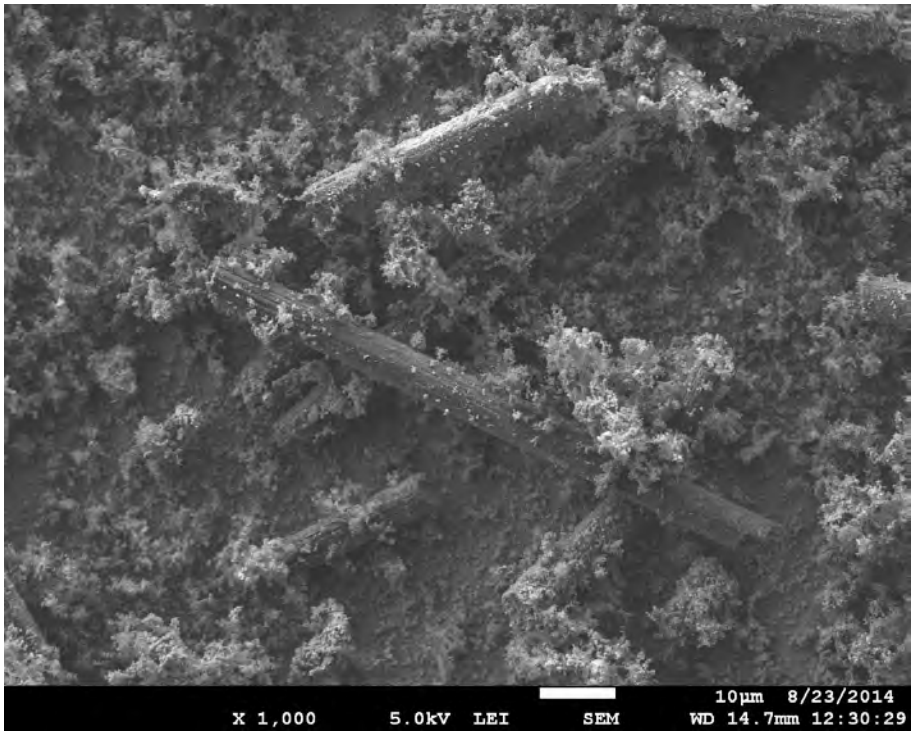


Witness plate is completely covered in black sooty debris – even protected areas under the Whipple plates.

Witness Plate Post Test : Aluminum Disk D2 (protected)

Secondary Electron SEM Images (250X, 1KX)

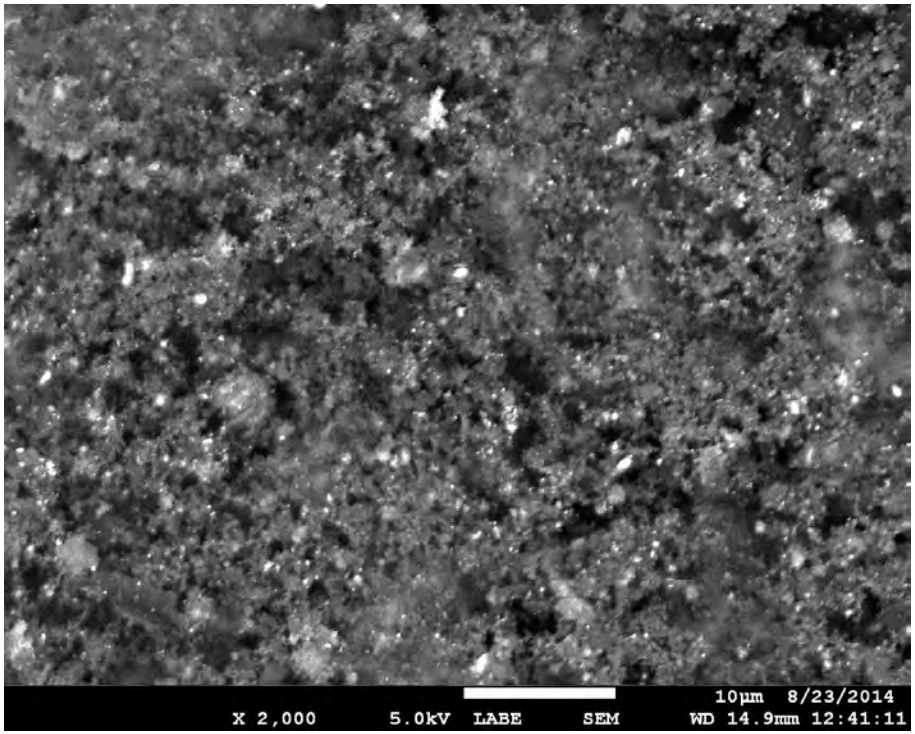


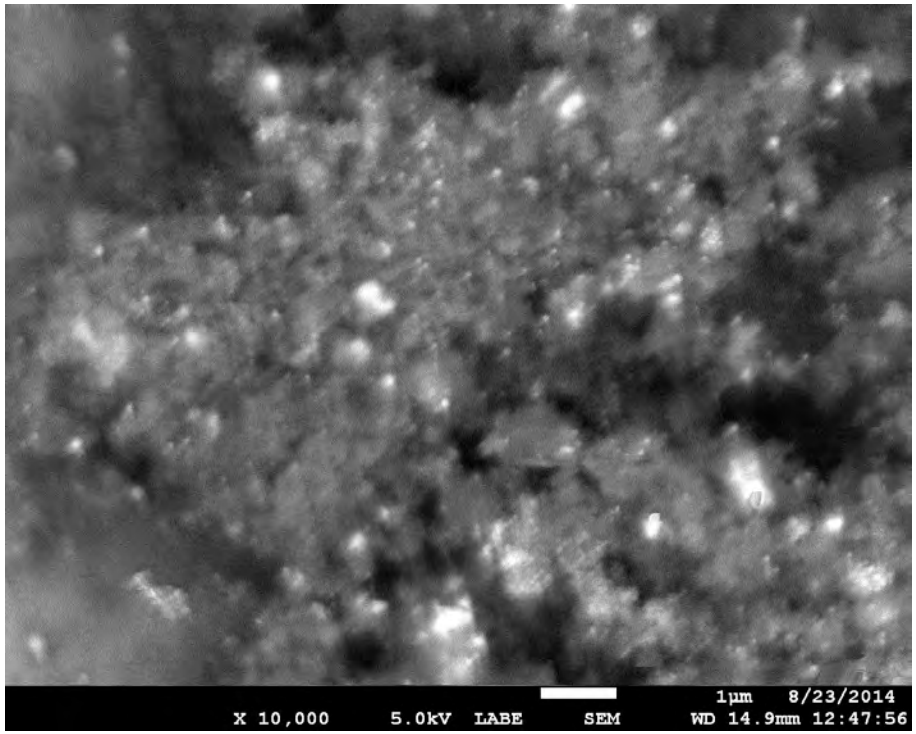


Note fragments of carbon fibers.

Witness Plate Post Test: Aluminum Disk D2 (protected)

Backscatter Electron SEM Images (2KX, 10KX)

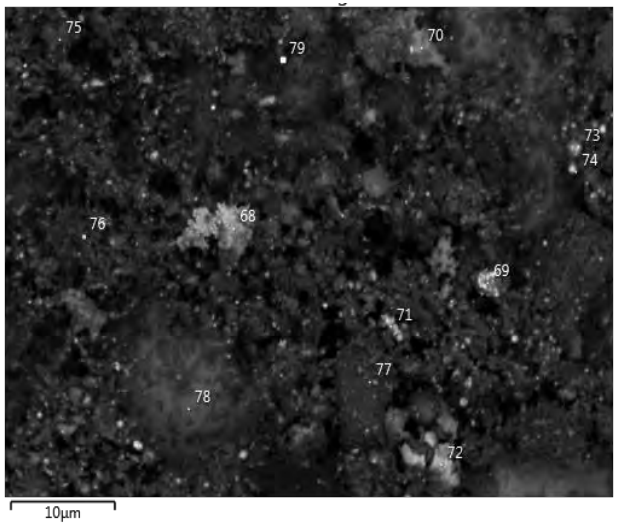


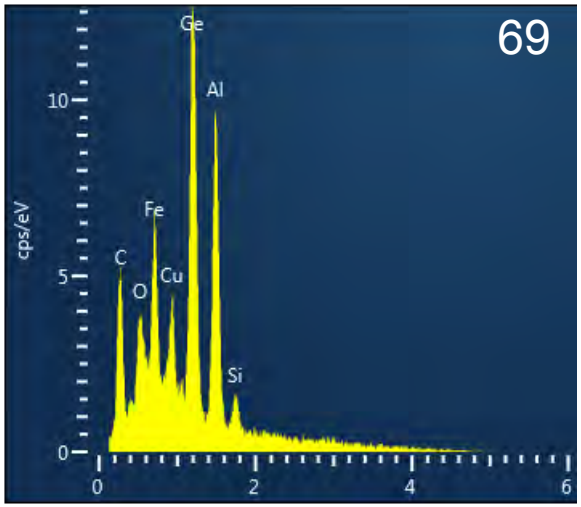


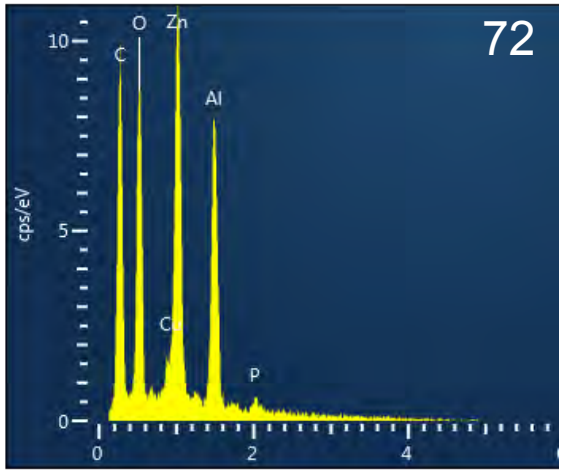
Agglomerates of nano carbonaceous material and solidified molten droplets of higher Z material

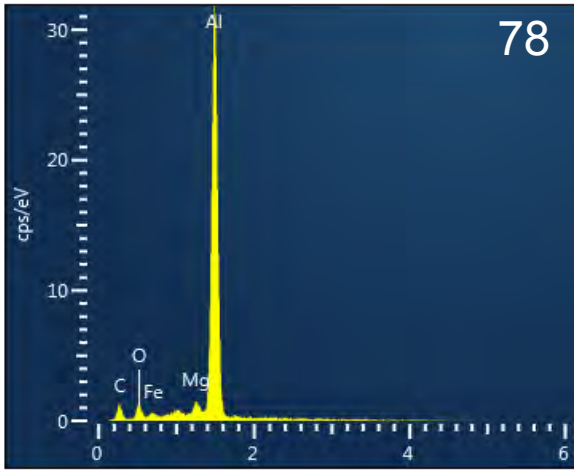
Witness Plate Post Test : Aluminum Disk D2 (protected)

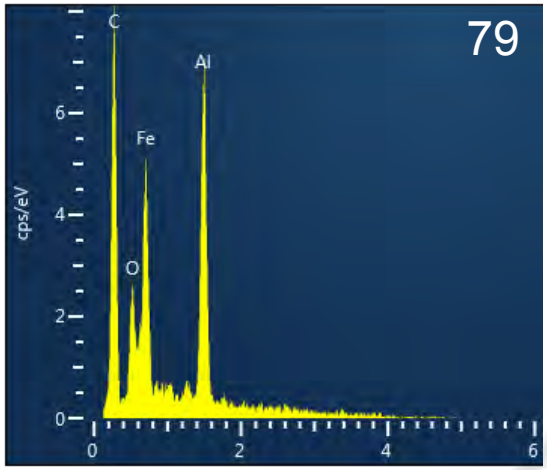
SEM EDS Spectra (5KV)





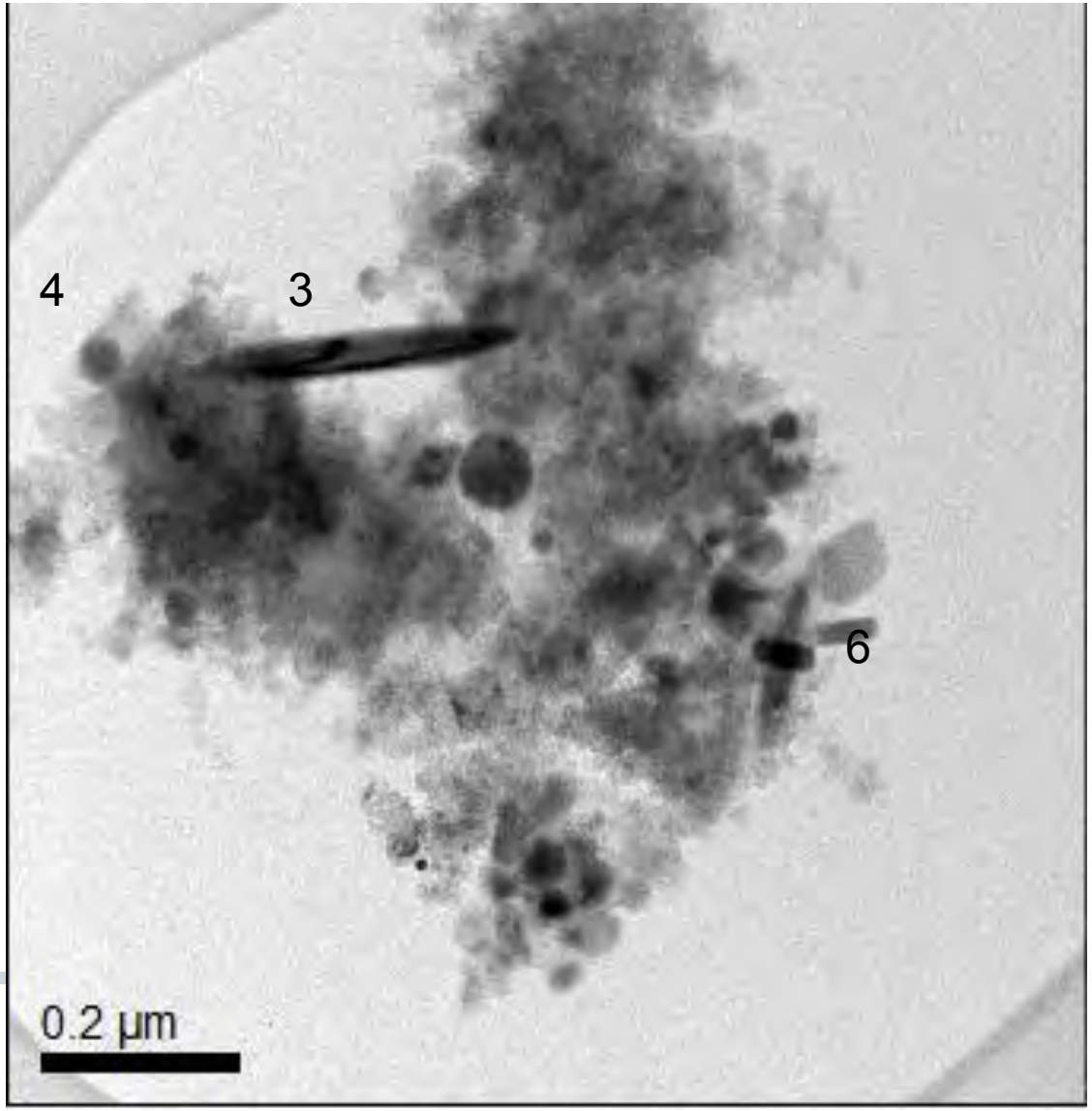






Al, C, Cu, O, Fe, Zn and Ge are common.

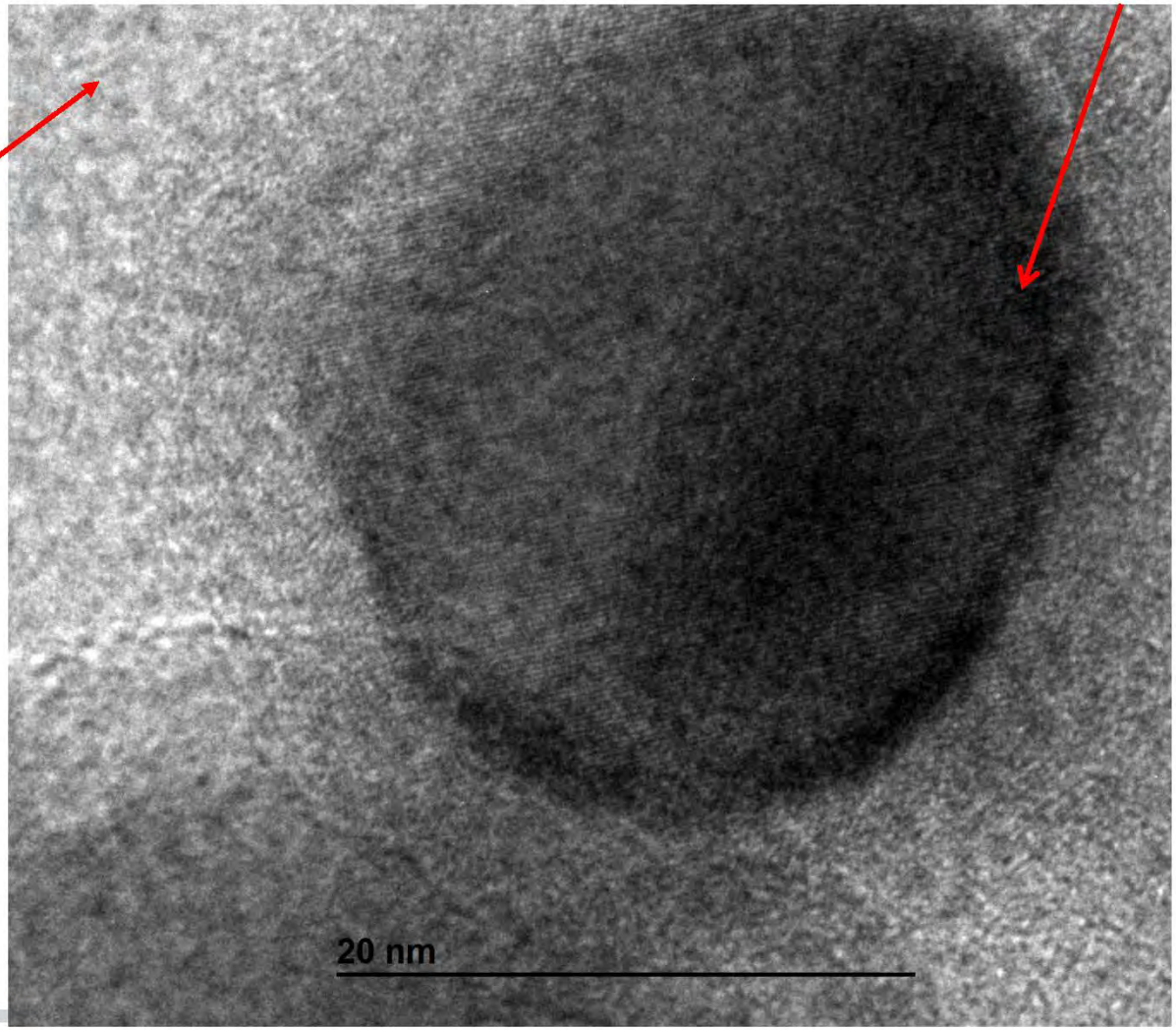
Witness Plate Al Disk D2: Loose Material on TEM Grid: BF TEM Location 4



Witness Plate Post Test D2: Area 2 BF-TEM

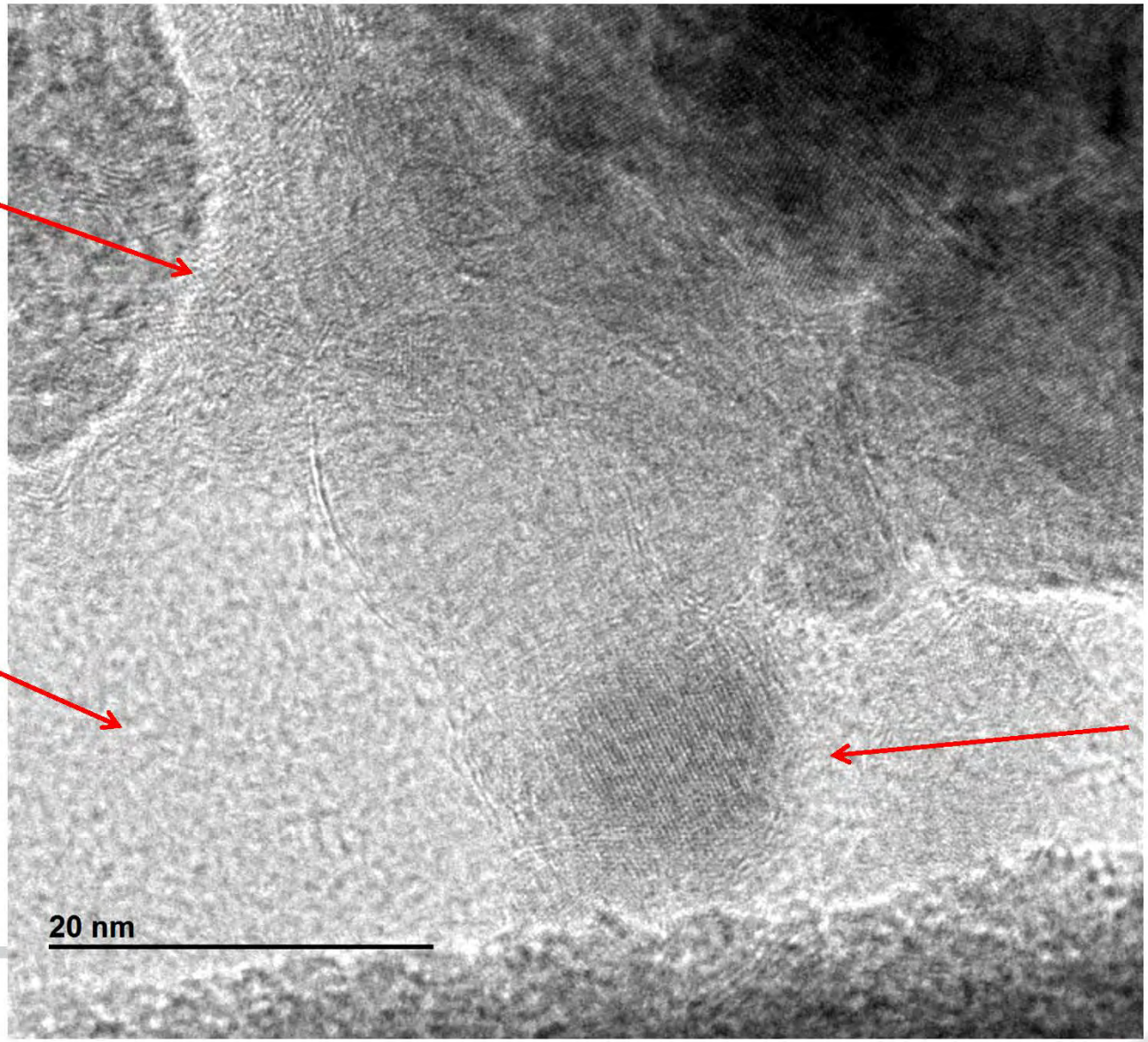
Crystalline lattice fringes

Amorphous C support film

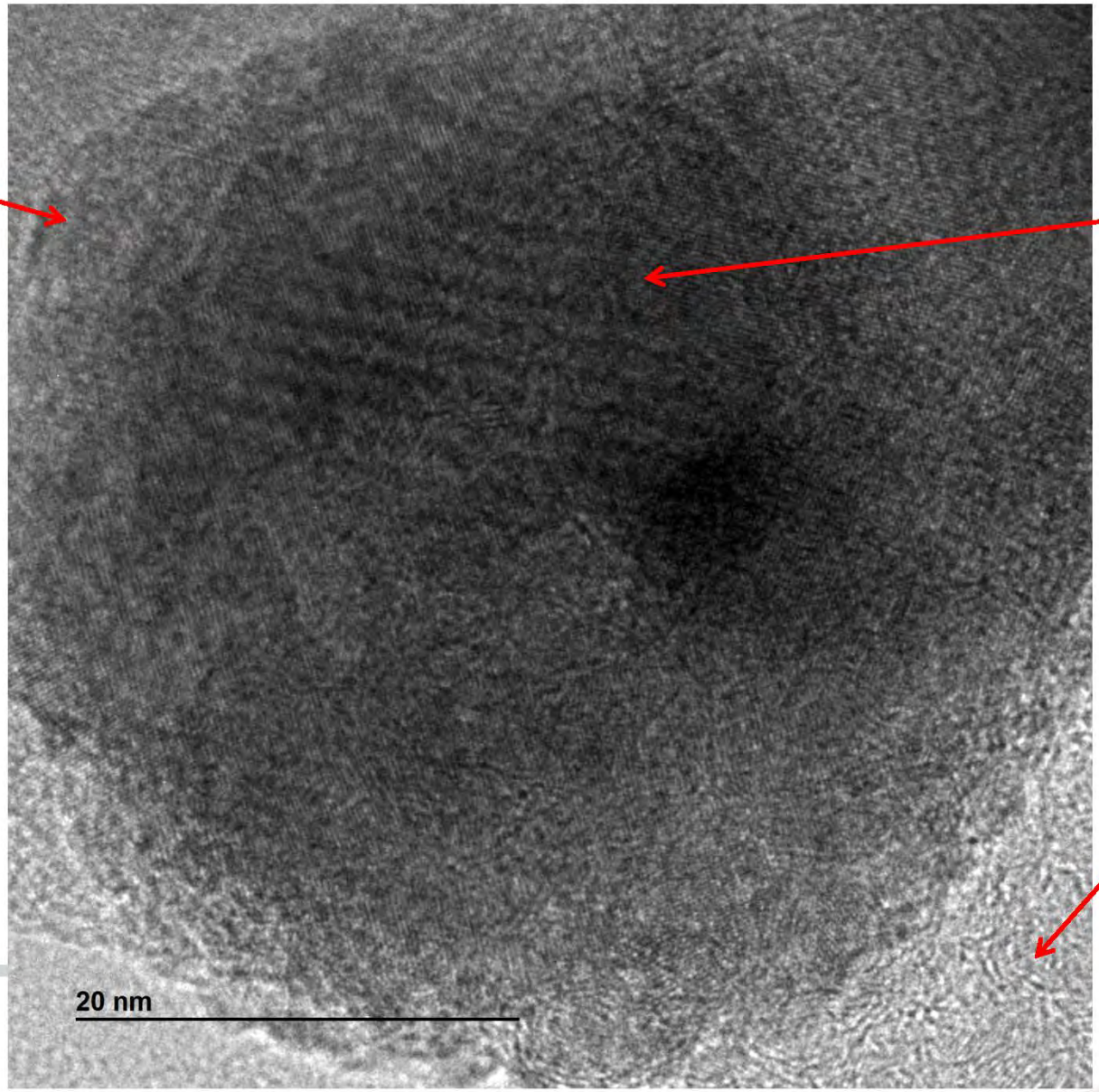


Particle is nominally a single crystal or consists of only a few crystalline grains.

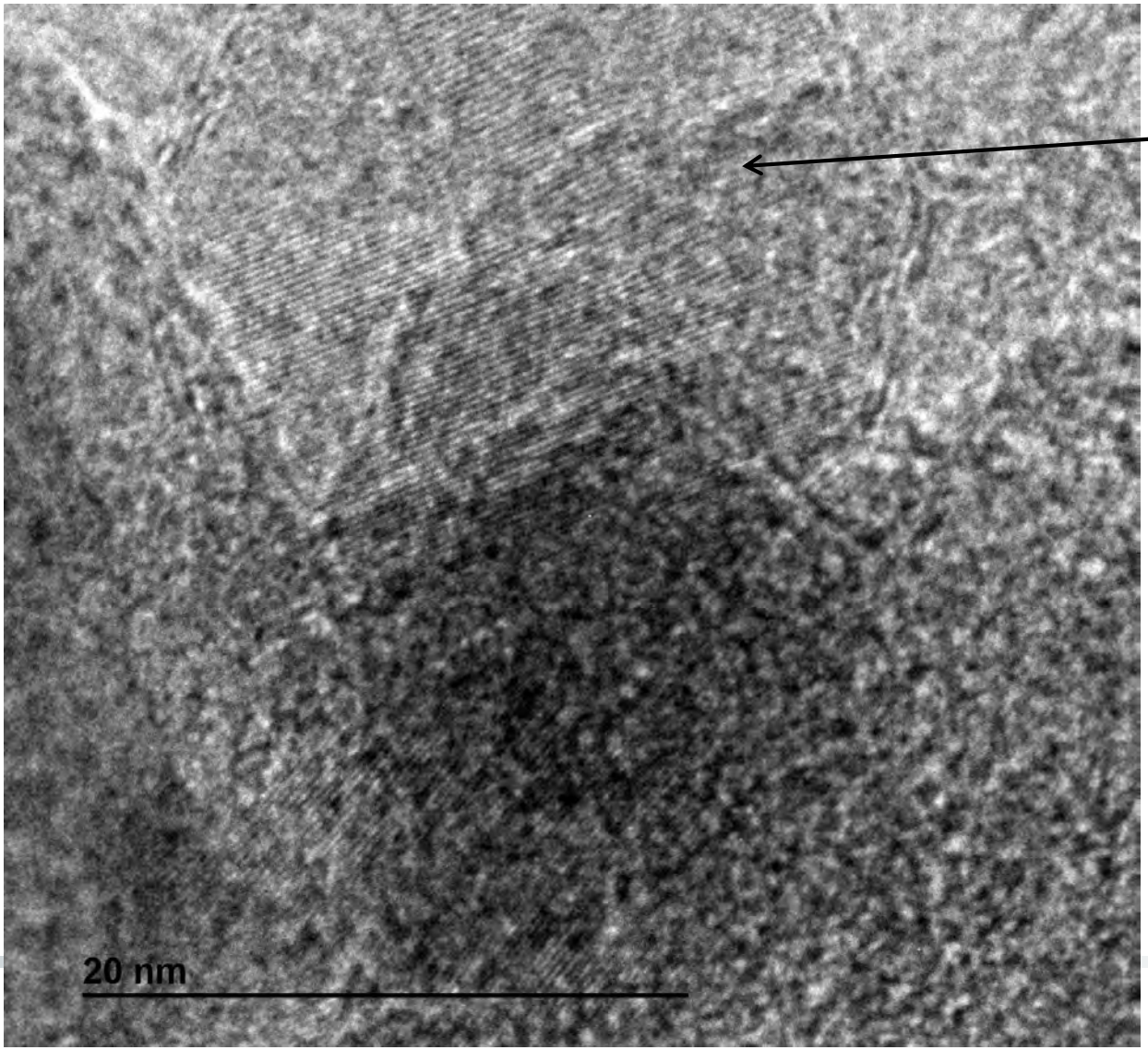
Witness Plate Al Disk D2: Location 4 Area 3 BF-TEM



Witness Plate Al Disk D2 : Location 4 Area 4 BF-TEM



Witness Plate Post Test: Al Plate D2: Area 4 BF-TEM

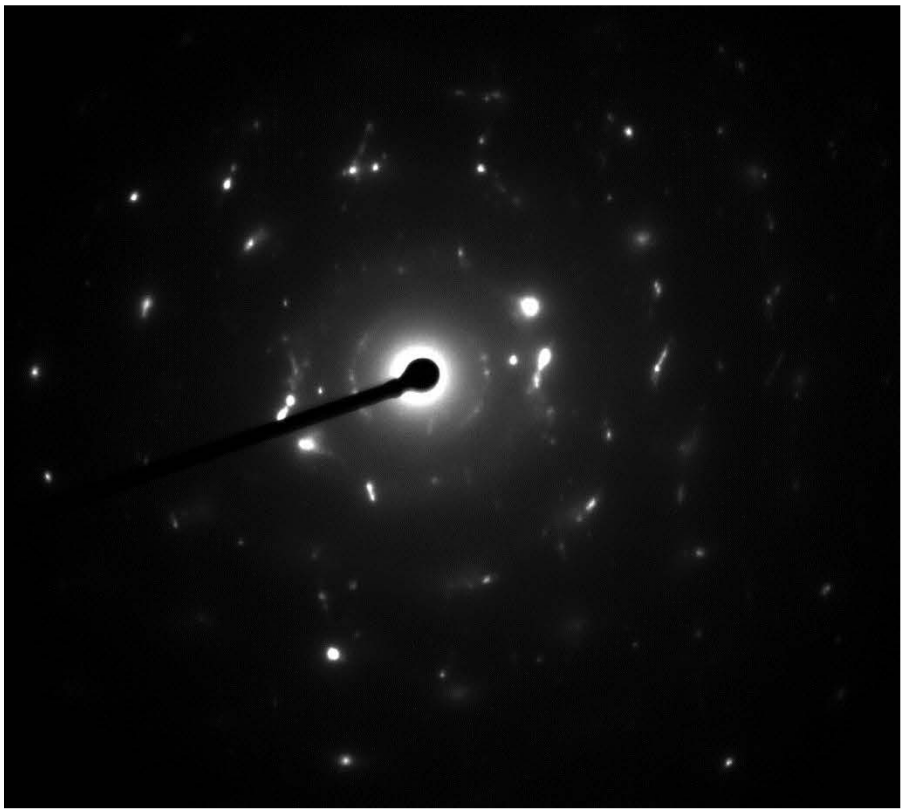


Crystalline lattice fringes

Particle is nominally a single crystal

SBU Marking

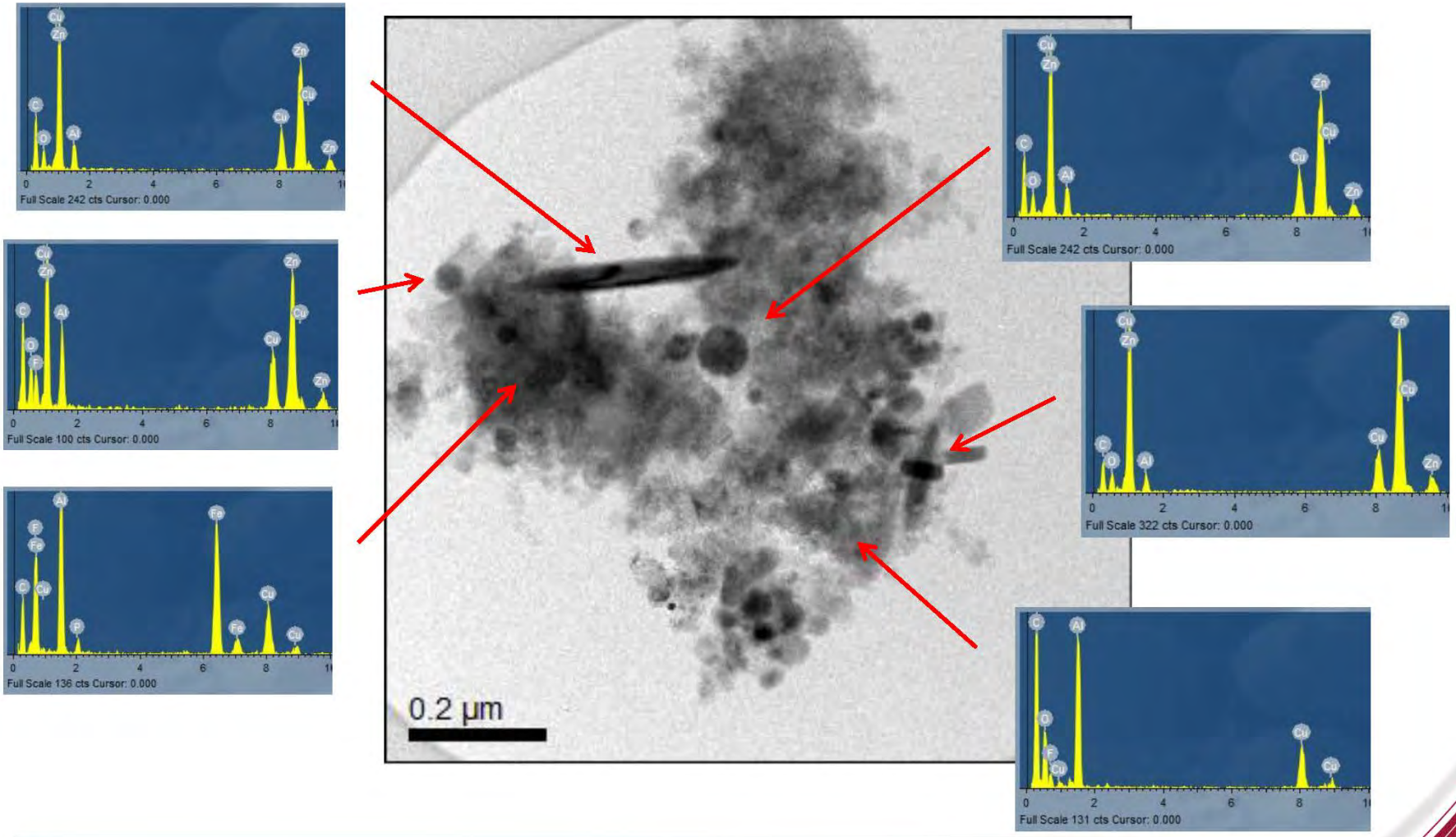
Witness Plate Al Disk D2 : Location 5 TEM Electron Diffraction



Spot patterns indicate crystallinity

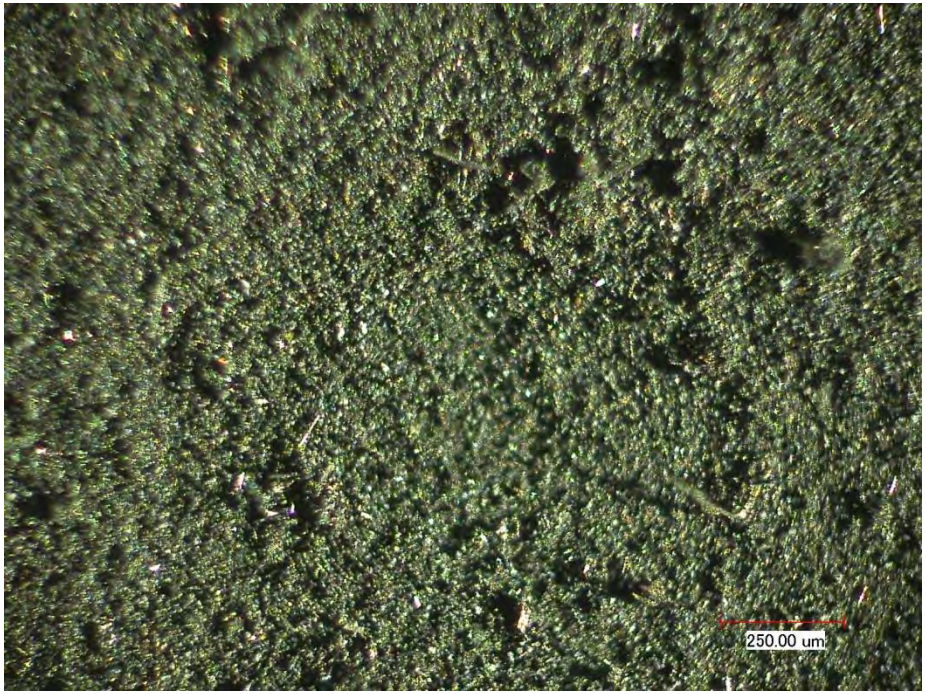
Patterns cover about 0.7 micron diameter area

Witness Plate Al Disk D2 : Location 4 TEM-EDS

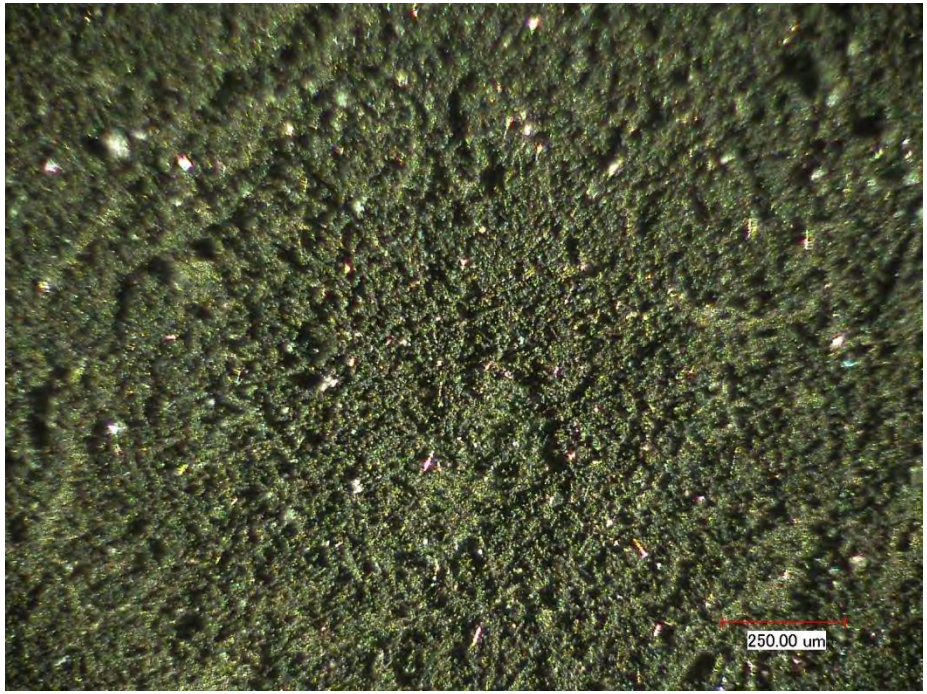


Discreet crystalline particles contain Zn. Al and Fe also detected.
Cu is probably from X-rays scattered from the microscope pole piece.

Witness Plate Post Test : Aluminum Disks
(Optical Images – 150X)



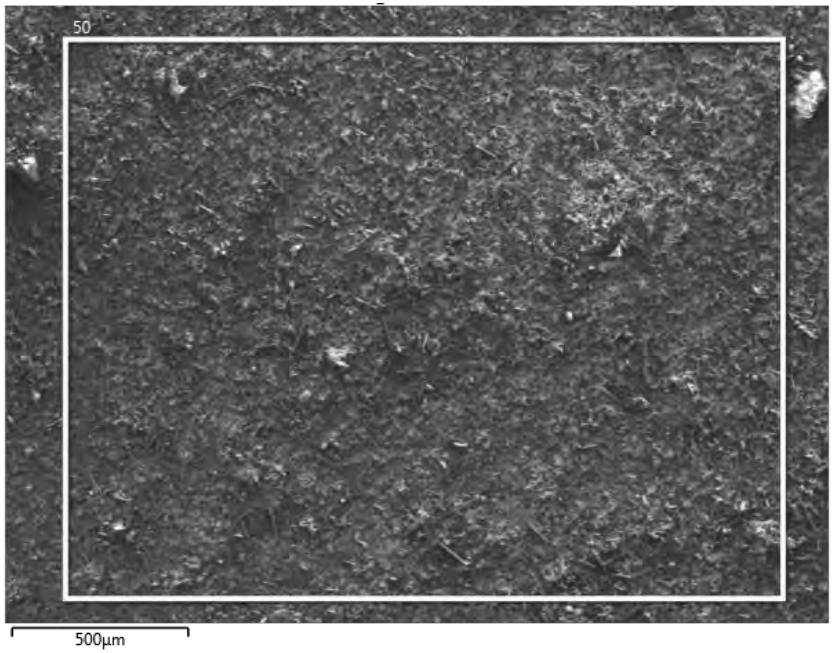
Exposed (B2)



Protected (D2)

Witness Plate Post Test : Aluminum Disk B2 (exposed)

SEM EDS Average Composition (10KV)

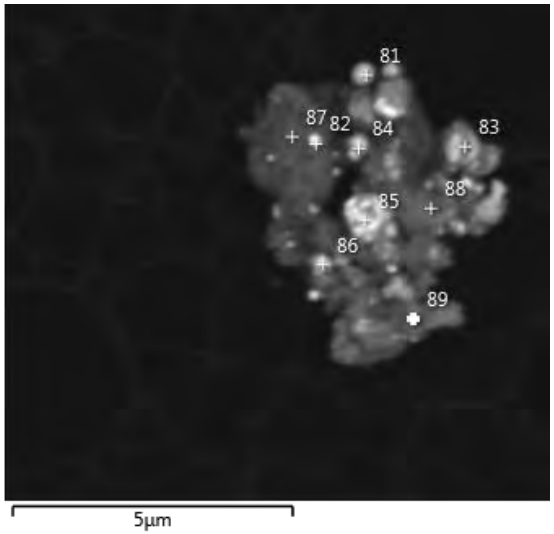


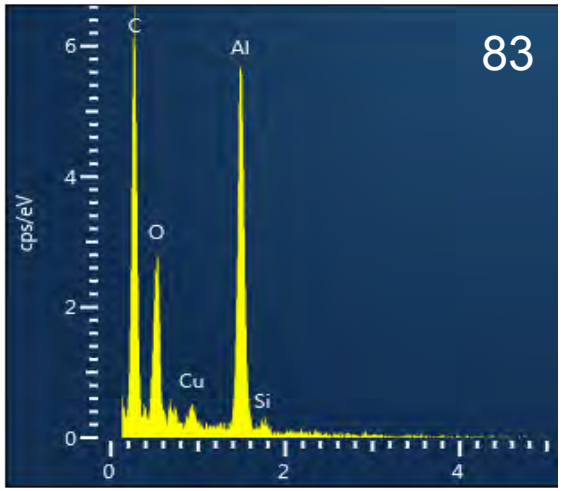
	Atomic %	Atomic %
C	63.67	64.82
O	15.89	15.58
F	1.22	1.61
Na	0.00	0.00
Mg	0.22	0.27
Al	13.74	12.72
Si	0.46	0.43
P	0.34	0.41
S	0.07	0.06
Ca	0.00	0.00
Cr	0.34	0.35
Fe	1.51	1.29
Cu	2.11	1.83
Zn	0.00	0.00
Ge	0.37	0.47
Ag	0.08	0.11
Sn	0.00	0.07
Total	100.00	100.00

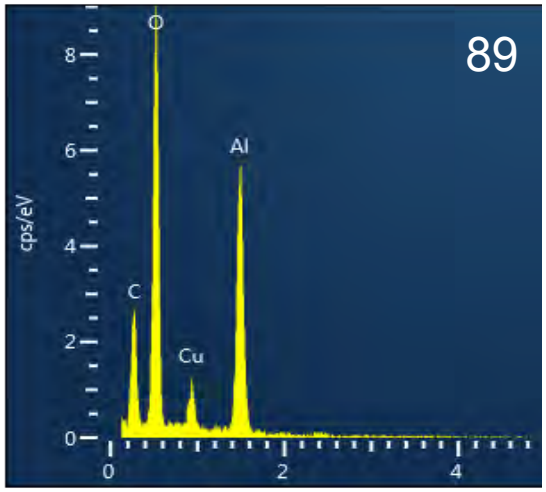
Elemental abundances are similar to SEM stub witness plates on soft catch panels.

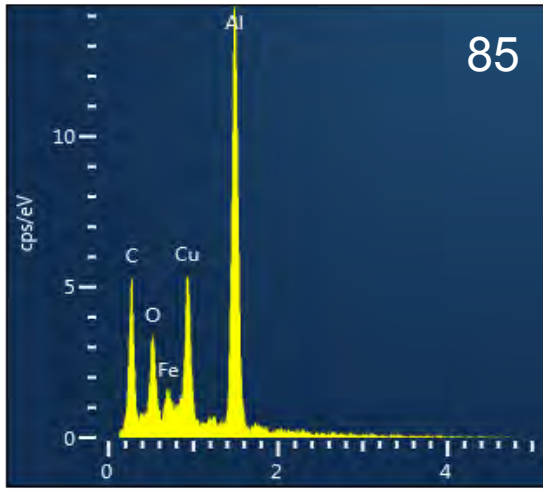
Witness Plate Post Test : Aluminum Disk B2 (exposed)

SEM Images of particles suspended on lacey carbon films on Cu TEM grid



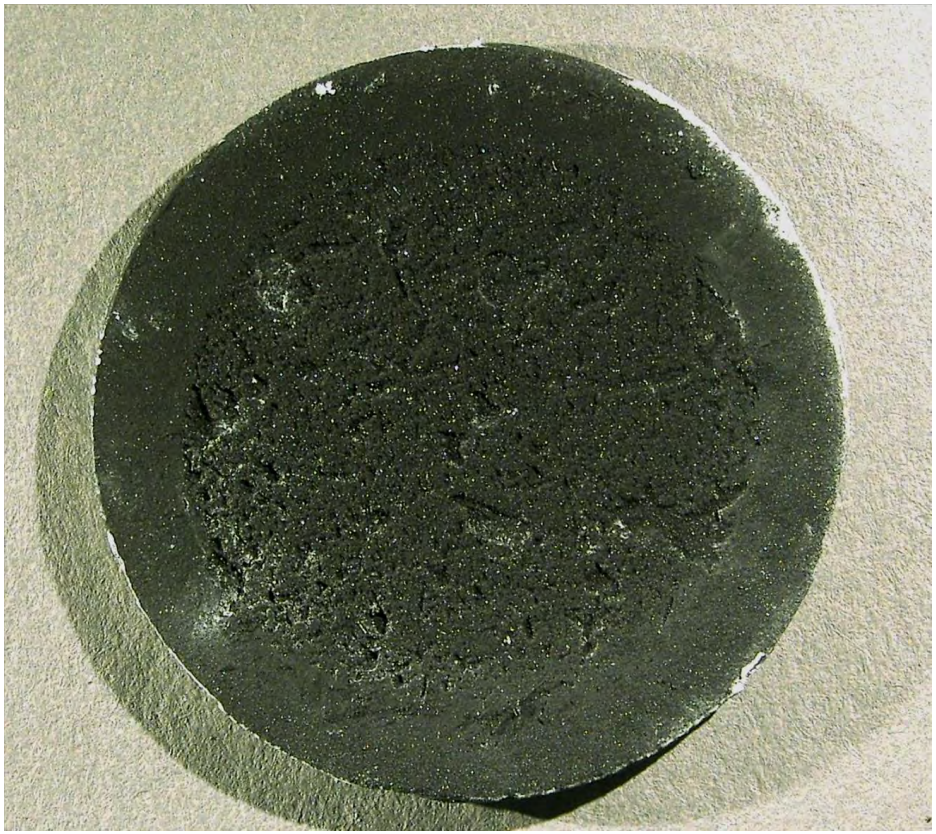




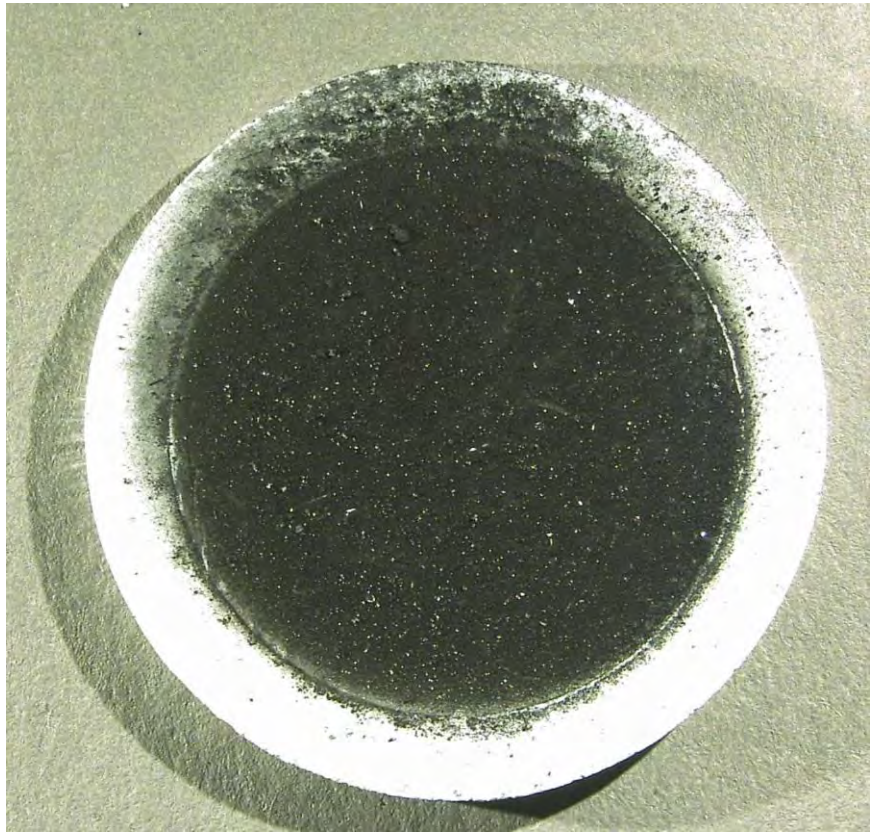


Al, C and O the are most common constituents, also minor Cu.

Witness Plate Post Test: Z93 Painted Aluminum Disks



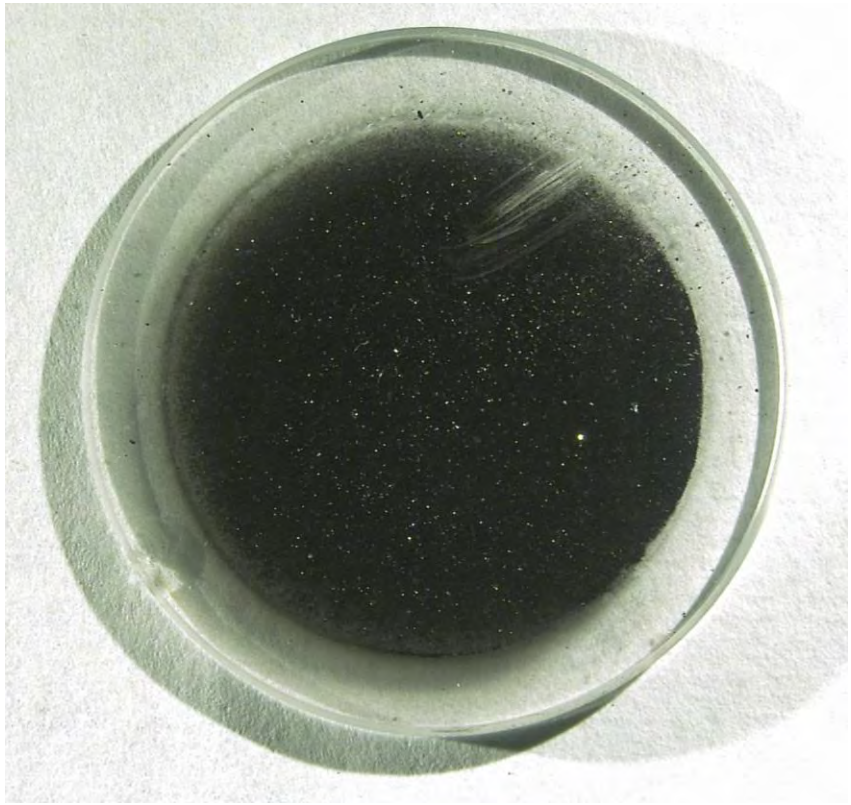
Exposed



Protected

Z-93 paint consists of ZnO in a potassium silicate binder

Witness Plate Post Test : Fused Silica Disks

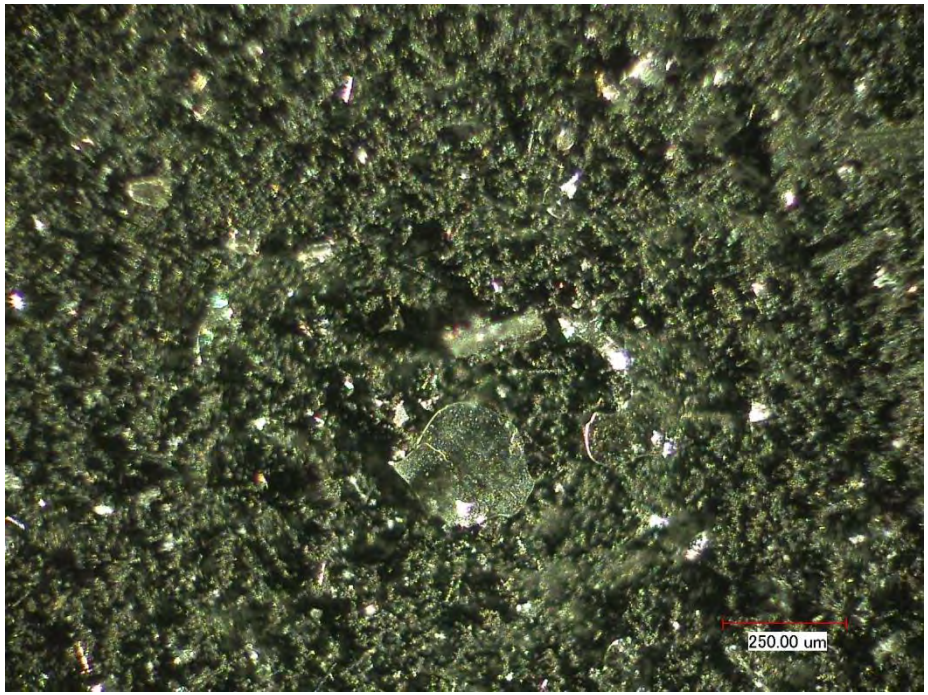


Protected (C1)

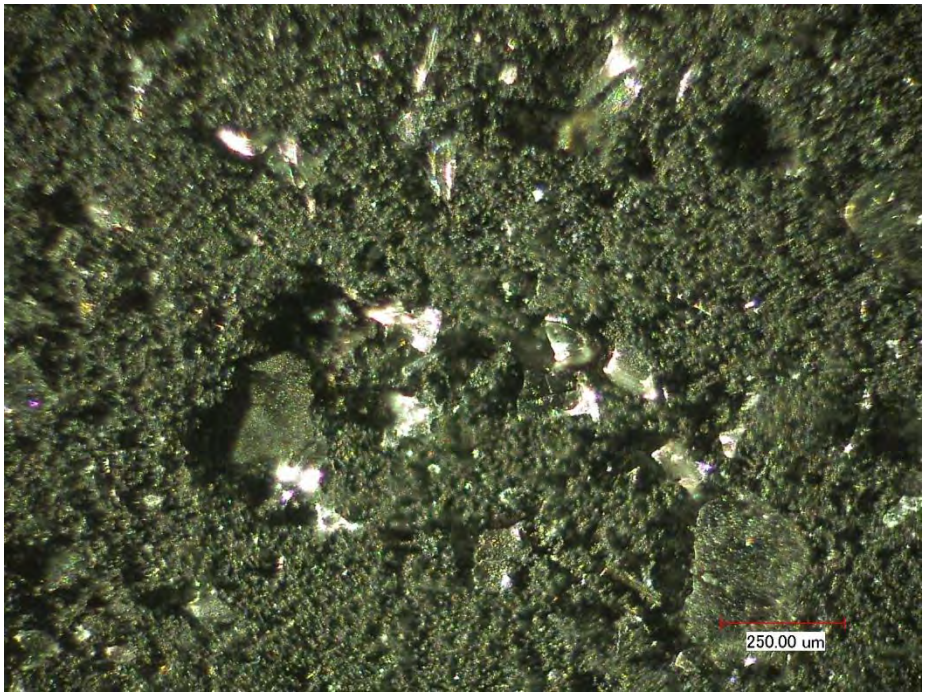
Protected (D1)

The directly exposed disks were destroyed

Witness Plate Post Test : Fused Silica Disks –
Optical Images (150X)



Protected (C1)

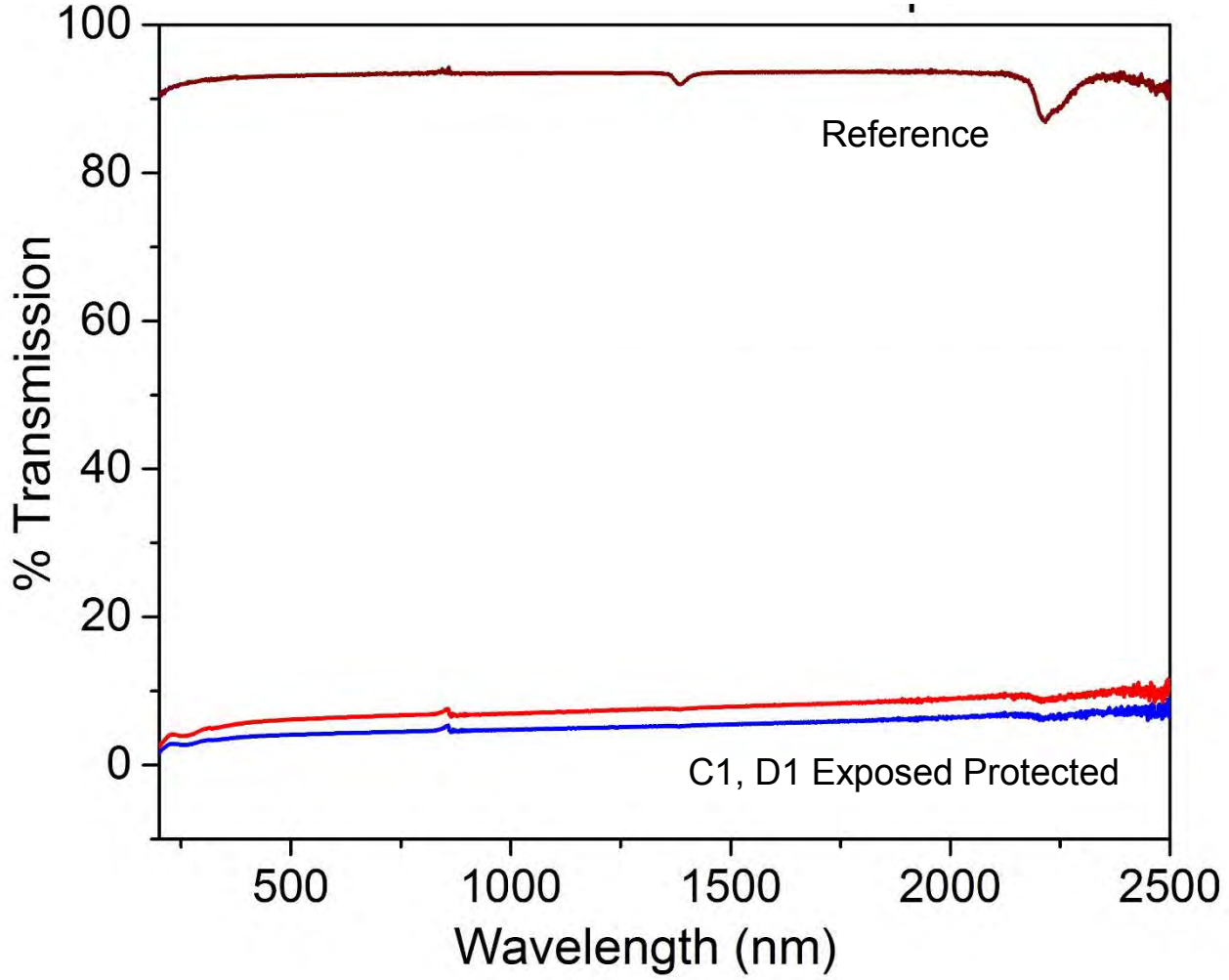


Protected (D1)

Clear angular fragments are soft catch foam debris.

Witness Plate Fused Silica Disks

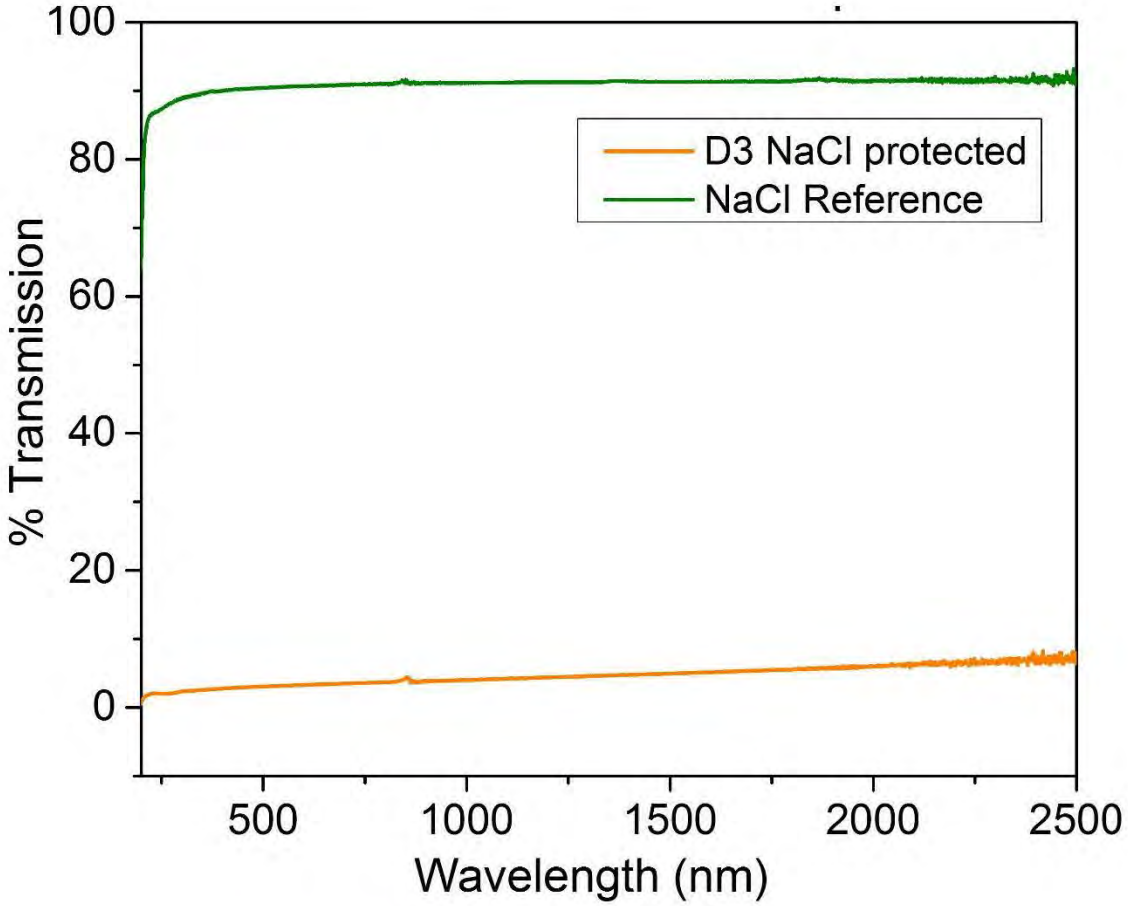
UV-VIS-NIR Transmission



Significant decrease in transmission from 94% to 8%.

Witness Plate NaCl Disk

UV-VIS-NIR Transmission



Significant decrease in transmission from 90% to 12%.

Appendix 2

SBU Marking

Technical Reports Addendum Asset Summary

AEROSPACE

TRAAS ID #: 20150114064543148 20

Report Name: DebriSat Laboratory Analyses

Aerospace Report Number: TOR-2014-00876

Start Date of Test: 2014-05-01 Created By: 14820 Adams, Paul M JO: 850672

End Date of Test: 2014-12-31 First Aerospace Author / PI: 14820 Adams, Paul M

Program: DebriSat

Description:

Keywords:

Asset: AAA123 Manufacturer: PERKIN ELMER CORPORATION Model: LAMBDA 900 Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2013-04-17	2014-08-17	TMT-NORMAL	fa6f32f7eed80b468d87b46e4dfec1aa
2014-08-18	2016-03-13	TMT-NORMAL	777773c04920e64b8f107f76514d6a40

Asset: AAA126 Manufacturer: PERKIN ELMER CORPORATION Model: PELA-1000 Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2013-04-17	2016-04-17	TMT-NORMAL	ed846df747a19149bb42006784ea95a7

Asset: ABH857 Manufacturer: RENISHAW Model: INVIA Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2013-07-18	2014-07-13	TMT-NORMAL	fd9ec878f3a7f246a89e074b2d36e529
2014-07-16	2200-01-01	SAE	e6467a74b10adc47b9ae2e6d00f01835
2014-07-16	2015-12-13	TMT-NORMAL	af200cfb9e350842a2f66701f5d97550

SBU Marking

Asset: ABN670 Manufacturer: JEOL (USA) INC. Model: JEM3100F Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2013-01-09	2014-05-04	TMT-NORMAL	885d0189ca15864aba20d3b3e6d5eb0a
2014-05-08	2015-12-06	TMT-NORMAL	0f70eaaaf9351b42901343965adb1738

Asset: ABW501 Manufacturer: THERMO-NICOLET Model: 6700 Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2014-03-17	2015-08-16	TMT-NORMAL	c0ad25e310e49243ae8a043b67c2c0f1

Asset: ACR364 Manufacturer: JEOL (USA) INC. Model: JSM-7600F Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2014-02-05	2015-07-05	TMT-NORMAL	d498d9360ad1224f85d04309c6869674

Asset: ACR429 Manufacturer: OXFORD INSTRUMENTS Model: X-MAX Usage Start Date: 2014-05-01 Usage End Date: 2014-12-31 Asset Comment:

Date:	Calibration Due Date:	Comment:	Certificate Number:
2013-02-08	2014-06-08	TMT-NORMAL	106f65ed9dfc8043869a5da06587e7fa
2014-06-02	2015-12-27	TMT-NORMAL	f840182f4f92e2449d541c92c3465ec2

*Support and Auxiliary Equipment are not calibrated.

AEROSPACE REPORT NO.
TOR-2015-00876

DebriSat Laboratory Analyses

Approved Electronically by:

Frank L. Knight, SYSTEMS DIRECTOR
ENGINEERING
ENGINEERING DIRECTORATE
OFFICE OF EVP/SSG

David J. Gorney, EXECUTIVE VP
OFFICE OF EVP/SSG

Technical Peer Review Performed by:

Shant Kenderian, DIRECTOR DEPT
MATERIALS PROCESSING DEPT
SPACE MATERIALS LABORATORY
ENGINEERING & TECHNOLOGY GROUP

Steven C. Moss, DIRECTOR DEPT
MICROELECTRONICS TECHNOLOGY
DEPT
ELECTRONICS & PHOTONICS
LABORATORY
ENGINEERING & TECHNOLOGY GROUP

SY0008

External Distribution

REPORT TITLE
DebriSat Laboratory Analyses

REPORT NO.	PUBLICATION DATE	SECURITY CLASSIFICATION
TOR-2015-00876	May 4, 2015	UNCLASSIFIED

Thomas Huynh
SMC/ENC
thomas.huynh@us.af.mil

Mitch Nolen
AEDC
mitchell.nolen.ctr@us.af.mil

Jesse Edwards
SMC/ENC
jesse.edwards.4@us.af.mil

Brian Roebuck
AEDC
brian.roebuck.ctr@us.af.mil

J.-C. Liou
NASA-JSC
jer-chyi.liou-1@nasa.gov

John Opiela
NASA-JSC
john.n.opiela@nasa.gov

Heather Cowardin
NASA-JSC
heather.cowardin@nasa.gov

APPROVED BY (AF OFFICE) Thomas T. Huynh DATE 18 Feb 2015